



## **A new numerical framework to simulate viscoelastic free-surface flows with the finite-volume method**

**Comminal, Raphaël; Spangenberg, Jon; Hattel, Jesper Henri**

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**ic-rmm1**

**The 1<sup>st</sup> International Conference on  
Rheology and Modeling of Materials**

# **BOOK OF ABSTRACTS**

**2013.**

# **The 1<sup>st</sup> International Conference on Rheology and Modeling of Materials**

Miskolc-Lillafüred, Hungary  
October 7-11, 2013

## **BOOK OF ABSTRACTS**

Edited by  
László A. GÖMZE



**ic-rmm1**

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Prof. Dr. László A. Gömze  
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## **PREFACE**

Understanding the rheological properties of materials and their rheological behaviors during their manufacturing processes and in their applications in many cases can help to increase the efficiency and competitiveness not only of the finished goods and products but the organizations and societies also. The more scientific supported and prepared organizations develop more competitive products with better thermal, mechanical, physical, chemical and biological properties and the leading companies apply more competitive knowledge, materials, equipment and technology processes. The idea to organize in Hungary the 1<sup>st</sup> International Conference on Rheology and Modeling of Materials we have received from prospective scientists, physicists, chemists, mathematicians and engineers from Asia, Europe, North and South America including India, Korea, Russia, Turkey, Estonia, France, Italy, United Kingdom, Chile, Mexico and USA.

The goals of **ic-rmm1** the 1<sup>st</sup><sup>nd</sup> International Conference on Rheology and Modeling of Materials are the following:

- Promote new methods and results of scientific research in the fields of modeling and measurements of rheological properties and behavior of materials under processing and applications.
- Change information between the theoretical and applied sciences as well as technical and technological implantations.
- Promote the communication between the scientist of different disciplines, nations, countries and continents.

The international conference **ic-rmm1** provides a platform among the leading international scientists, researchers, PhD students and engineers for discussing recent achievements in measurement, modeling and application of rheology in materials technology and materials science of liquids, melts, solids, crystals and amorphous structures. Among the major fields of interest are the influences of material structures, mechanical stresses, temperature and deformation speeds on rheological and physical properties, phase transformation of foams, foods, polymers, plastics and other competitive materials like ceramics, nanomaterials, medical- and biomaterials, cosmetics, coatings, light metals, alloys, glasses, films, composites, hetero-modulus, hetero-viscous, hetero-plastic complex materials, petrochemicals and hybrid materials, ... etc.

Multidisciplinary applications of rheology and rheological modeling in material science and technology encountered in sectors like alloys, ceramics, glasses, thin films, polymers, clays, construction materials, energy, aerospace, automotive and marine industry. Rheology in food, chemistry, medicine, biosciences and environmental sciences are of particular interests.

In accordance to the program of the conference **ic-rmm1** more than 160 inquiries and registrations were received from 51 countries. Finally the scientists and researchers have arrived to our conference from 42 countries. Including co-authors, the research work of more than 300 scientists are presented in this book.

Prof. Dr. László A. Gömze

*chair, ic-rmm1*



## **CONTENTS**

Plenary lectures	10
Session 1. Applied Rheology and Modeling of Clays, Minerals and Rocks	25
Session 2. Applied Rheology of Polymer Melts	35
Session 3. Applied Rheology and Material Structure of Foods	45
Session 4. Avalanche Dynamics in Rheology and Phase Transitions	54
Session 5. Experimental Study and Modeling of Complex Flow	61
Session 7. Multiscale Mechanics of Materials	68
Session 8. Rheological Fluid Mechanics	78
Session 9. Rheology and Modeling of Hetero-modulus Hetero-viscous Complex Materials	86
Session 12. Rheology of Biological Systems	89
Session 13. Rheology of Construction Materials and Concretes	94
Session 14. Rheology of Emulsions, Suspensions and Foams	101
Session 15. Rheology of Ferrofluids and Magneto-rheological Materials	114
Session 16. Rheology of Fluid Interfaces and Modeling	121
Session 17. Rheology of Melted Metals and Glasses	123
Session 18. Rheology of Nanofluids	127
Session 19. Rheometric Measurement – Methods, Equipment and	135
Session 21. Rheology of Drug and Cosmetic Materials	143

## **PLENARY LECTURES**

## PLENARY 1.

### Preparation of Functional Composite Materials Based on Chemically Derived Graphene using Solution Process

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Graphene-based materials have attracted enormous attention in both academia and industries because of their outstanding chemical and physical properties compared to conventional carbon based materials. Chemically derived graphenes such as graphene oxides (GOs) or graphene nanoplatelets (GNPs) are, in particular, promising building blocks for carbon-based functional composite materials. Tailored assembly of GOs/GNPs onto different substrates offers feasible routes towards the ultimate utilization of graphitic carbon materials in nanoelectronics, energy storage/conversion devices including flexible devices.

In this work, GOs and GNPs were assembled into functional composite materials using solution process from stable solvent dispersion. Based on the self-assembly principles of colloidal particle dispersion, three-dimensional (3D) macroporous structure of reduced GOs was fabricated by using polystyrene colloidal particles as a sacrificial template. To reduce the surface resistivity of macroporous structure, GNP thin film with GOs as a surfactant and a film stabilizer was coated on the both side of the prepared 3D structure. Due to the 3D continuous conductive pathway of reduced GOs, the prepared composite films show excellent electrical conductivities at relatively lower contents of conductive filler.

We also successfully developed foldable electronic circuits for paper substrates using vacuum filtration of GNP dispersion and a selective transfer process with a pen. Without need for special equipment, this method enables us to obtain graphene circuits on a paper substrate with controlled dimensions including width and thickness. The employed surfactant PSS was effective both for dispersion stability and the formation of a hydrophilic surface on the GNPs, which enabled a clear selective transfer process and favorable adhesion between the graphene and the paper substrate. The electronic circuits revealed a small change in conductance under various folding angles and maintained an electronic path on the paper substrate after repetitive folding and unfolding. Employing the graphene circuits, finally, we demonstrated foldable circuit boards based on paper substrates for operating LED chips under various kinds of deformation.

We believe that our approach could provide a meaningful method for a simple patterning of the dispersion-based graphene conductor to realize highly potential applications based on conductive paper-like structures.

**Keywords:** *graphene-based materials, graphene oxide, graphene nanoplatelet, self-assembly, 3D macroporous structure, paper electronics*

## PLENARY 2.

### Intriguing Nonlinear Instability Dynamics in Film Blowing Process

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The film blowing is one of the most important polymer processing operations widely used for producing bi-axially oriented film products in a single-step process. It has been in operation over 40 years and many researchers around the world have been seeking ways how to enhance the productivity and product film quality of this convenient process even further. All these efforts have been possible ever since the seminal work by Pearson and Petrie in 1970 who established the theoretical framework for modeling and simulation of the process. While two fundamentally different instabilities have been found in film blowing during the last four decades by various research groups, i.e., draw resonance and helical instability, transient simulation of these instabilities have been rather elusive due to the highly nonlinear character of the process dynamics generated by the complicated set of governing equations comprising hyperbolic partial differential equations. It is rather recent that we have successfully produced the first simulation results of film blowing (Hyun et al. 2004 and Lee et al. 2011) which closely portray the experimentally observed draw resonance instability. Now, we are in the process of simulating the other nonlinear instability of film blowing, i.e., helical instability, and hereby report the first simulation results which agree well with the experimental data, obtained by incorporating the buckling instability theory into the modeling of the helical instability.

**Keywords:** *film blowing, nonlinear dynamics, draw resonance, helical instability*

### PLENARY 3.

#### **Modeling the Statistical Properties of Acoustic Emission During the Compression of a Porous Material**

Xavier Illa<sup>1</sup>, Jordi Baró<sup>1</sup>, Álvaro Corral<sup>2</sup>, Antoni Planes<sup>1</sup>, Ekhard K.H. Salje<sup>3</sup>, Wilfried Schranz<sup>4</sup>, Daniel E. Soto-Parra<sup>5</sup>, and Eduard Vives<sup>1</sup>  
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It has long been stated that there are profound analogies between fracture experiments and earthquakes; however, few works attempt a complete characterization of the parallels between these so separate phenomena. We study the acoustic emission events produced during the compression of Vycor ( $\text{SiO}_2$ ). The Gutenberg-Richter law, the modified Omori's law, and the law of aftershock productivity hold for a minimum of 5 decades, are independent of the compression rate, and keep stationary for all the duration of the experiments. The waiting-time distribution fulfills a unified scaling law with a power-law exponent close to 2.45 for long times, which is explained in terms of the temporal variations of the activity rate.

The epidemic-type aftershock (ETAS) model, which has been broadly used to simulate the seismic activity, is used here to model the compression of Vycor. Obtaining the model parameters from the experimental data, the ETAS model is able to generate a signal with a waiting-time distribution that fulfills the former unified scaling law.

**Keywords:** *Avalanches Dynamics, Porous Materials, ETAS Model*

## **PLENARY 4.**

### **Consideration on the Interpretation of a Contact Angle on a Real Surface from Detailed Derivations of Young's equation, Cassie-Baxter equation and Wenzel equation.**

Kwangseok Seo<sup>1</sup>, Minyoung Kim<sup>1</sup>, Rinbok Wi<sup>1</sup>, Hwayong Kim<sup>2</sup>, Do Hyun Kim<sup>1</sup>  
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The wetting property between liquids and solid has become important in the analysis of rheological systems with contact line as the dimensions of the system to be understood are reduced. The simplest method to determine the wetting property is just dropping a liquid drop on a solid surface. The liquid drop on a solid surface will spread to form a thin film (complete wetting) or have a spherical shape sectioned by the surface (incomplete wetting). The wetting property can be easily predicted from this shape of the drop in terms of a contact angle. Young's equation has been used as a basic theoretical equation over 200 years to quantify the wetting property. Cassie-Baxter equation and Wenzel equation have been used for a real surface. However, limitations in using these equations were neglected that many researchers used them widely without care. Gao and McCarthy suggested in 2007 that these equations fail to describe a contact angle of a real surface. But their conclusion was from the experimental observations and systematic explanation was not sufficient. Here, these equations will be derived in detail with simple mathematics from a thermodynamic point of view. It will be shown that the most important reason why these equations cannot describe a real surface with contact angle hysteresis is the basic assumption used in the derivation that triple contact line is free to move on the surface. Limitations of these equations and some considerations on a contact angle on a real surface will be discussed during the derivation of the equations. We expect that our interpretation will be helpful in analyzing the system where rheological properties are affected dominantly by the wetting phenomena.

**Keywords:** *Young's equation, Cassie-Baxter equation, Wenzel equation, Contact angle,*

**PLENARY 5.**

**A Simple Model for plastic deformation and the statistics of slip avalanches  
(serrations in the stress strain curves)**

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A basic micromechanical model for deformation of solids with only one tuning parameter (weakening) is introduced. The model can reproduce observed stress-strain curves, acoustic emissions and related power spectra, event statistics, and geometrical properties of slip, with a continuous phase transition from brittle to ductile behavior. Exact universal predictions for slip (serration) statistics and acoustic emissions are extracted using mean field theory and renormalization group tools. The results agree with recent experimental observations and simulations of related models for dislocation dynamics, material damage, and earthquake statistics.

**Keywords:** *Plasticity, Slip-Avalanches, Criticality, Serrations, Noise-Statistics*

## **PLENARY 6.**

### **Dynamics of Soft, Biological Interfaces**

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Biological systems are normally high-interface systems and these surfaces are laden with biological molecules that render them rheologically complex. The resulting nonlinearities with response to surface stresses and strain are often essential to their proper function and these are explored using recently developed methods that reveal interfacial moduli and microstructure. Three applications are discussed.

1. The tear film of the eye is a composite structure of an aqueous solution of protein and biomacromolecules. This thin layer is further covered by a film comprised of meibomian lipids excreted during each blink. The purpose of the meibum has been largely unexplained although one prevailing suggestion is that it suppresses evaporation. Recent measurements in our laboratory demonstrate that this layer is strongly viscoelastic and this property has dramatic effects on the dynamics of the moving contact line and stability against dewetting.

2. Biofilms are protective layers produced by bacteria colonies that offer protection against desiccation and external agents that can attack the colonies. This layer, a result of amyloid fiber produced by the bacteria make it difficult to treat intestinal tract infections in our own bodies and methods to monitor the kinetics of biofilm development and the resulting response of the films to excipient materials that might upregulate or quench amyloid production are needed. Experiments are described where interfacial moduli are demonstrated to be very effective in sensing the presence of these films and provide a convenient format for the systematic introduction of external, chemical agents.

3. Vascular endothelial cells line the interior walls of our blood vessels and are sensitive to surface shear stresses. These stresses are known to affect the shape and orientation of endothelial cells. It is evident that the spatial homogeneity of flow can affect vascular health and it is well-documented that lesions form in regions of high curvature, bifurcations, and asperities in blood vessels. Experiments are described where stagnation point flows are used to create regions of well controlled flow stagnation and spatial variation of wall shear stresses. Live-cell imaging is used to monitor the fate of cells attached to surfaces experiencing flow impingement and it is revealed that endothelial cells migrate and oriented in such flows to create remarkable patterns of orientation and cell densification.



## PLENARY 7.

### **Selection of appropriate polyoxymethylene based binder for powder injection moulding feedstock material**

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Polyoxymethylene copolymer (POM) is considered a high performance engineering polymer with many applications primarily in the automotive industry, due to its very good mechanical properties such as high modulus, stiffness, hardness, fatigue and creep resistance. Currently, POM has also found uses in powder injection molding (PIM) technology. PIM is one of the most versatile methods for the manufacturing of small complex shaped components from metal or ceramic powders for the use in many industries, like automotive, electronics and medical devices. PIM consists of mixing powders with a polymeric binder, injecting this mixture in a mould, debinding and then sintering. The main advantage of using POM in PIM technology is the faster debinding rates compare to polyolefin-based feedstock materials, since POM sublimates into its monomer directly when exposed to an acid vapour.

The feedstock material for PIM has two main contradictory requirements; on one hand it must have low viscosity in order to facilitate the injection molding process, but once the molded part solidifies it must have sufficient mechanical strength so that it does not break or deform during handling and debinding. POM-based feedstock materials have indeed superior mechanical strength in the solid state, but the main problem is their high viscosity, which can in fact difficult the injection moulding process. For this reason it was decided to prepare POM copolymers with different average molecular weights and investigate the effect of the average molecular weight on the viscosity and solid mechanical properties, in order to select the correct copolymer to be used as binder for PIM feedstock.

In this work, POM materials of different molecular weights have been prepared and characterized in terms of oscillatory viscosity and impact toughness. As expected, both properties increase as the average molecular weight increases. However, the Newtonian viscosity of POM increases with average molecular weight following a power law function, as it is the case with other linear entangled polymers. While the increase in impact toughness does not follow a simple relationship with molecular weight and it appears that there is a plateau at small molecular weights. With the information here gathered, it is possible to suggest that a POM copolymer with an average molecular weight of around 24000 g/mol could be used as the main component of a binder used for PIM. As compared to the currently available POM-based binder, using POM with the suggested molecular weight can lead to a decrease in viscosity of 200 times, while reducing toughness only by 10 times; this can be considered a significant improvement on the performance of POM-based binders for PIM.

**Keywords:** *polyoxymethylene copolymer, powder injection moulding, impact toughness, viscosity, molecular weight.*

## PLENARY 8.

### Optical coherence tomography velocimetry studies of complex fluids

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We will review recent progress made with the optical coherence tomography velocimetry technique to measure velocity profiles in a fluids rheometer<sup>1,2</sup>. This allows the velocity of 3.4 picolitre volumes of complex fluid to be probed inside a plate-plate rheometer of 1mL total volume. The initial infra red (1300 nm) fibre optic design using a Michelson interferometer has been replaced by a Mach Zender arrangement to provide improved signal to noise. Furthermore the introduction of a balanced detector and a data acquisition card with a higher sampling rate, again allows improvements to be made in the signal to noise ratio. Phase modulation of the Doppler shifted signal using an electro optical modulator, allows the effects of the low frequency 1/f noise to be reduced and enables the measurement of velocity profiles at lower shear rates. Shear banding and wall slip phenomena have been studied in polyacrylamide solutions, margarine, and tomato paste, as well as model hard sphere colloidal systems.

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<sup>2</sup>M.Harvey, T.A.Waigh, 'Optical coherence tomography velocimetry in controlled shear flow', *Physical Review E*, 2011, 83, 031502.

**Keywords:** OCT, shear banding, polymers, polyelectrolytes, colloids, foods

## **PLENARY 9.**

### **The Rheological basis of Bubble formation in cereal products**

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This presentation will integrate the work we have done on understanding bubble dynamics in mixing and extrusion during the last 20 years and in particular review previous work and report new science on the rheological basis of bubble formation. We have studied, nucleation, bubble growth, bubble break-up and related many of these phenomena to biaxial extensional flows and the extensional flow distribution in mixers and extruders. We also related bubble size distribution to the texture of foam-like cereal products. Lately Bubble break-up was studied during mixing of a viscous Newtonian fluid in a co-rotating twin screw continuous mixer using experimental observations and finite element method (FEM) simulation of dispersive mixing. The decrease in bubble break-up was found to be due to the decrease in the elongation flow density and an increase in local forward flow in the forward angle stagger configuration. Maximum stable bubble diameters and effective shear rate for break-up calculated at different locations in the mixer using the fundamental capillary number theory for drop and bubble break-up correlated well with the measured local mean bubble sizes and the mean local shear rates calculated using FEM simulations. Examining mixing efficiency is best done by mapping extension rate distribution in a mixer or extruder. Maximum extension rate values were seen in areas with high shear, between paddles and between paddle tips and the barrel wall. The highest extension rate values were seen between Manas- Zloczower mixing index  $\lambda$  values of 0.4 and 0.75, a much wider range than the predicted value for efficient dispersive mixing at  $\lambda=0.7$ . More elongational flow did not indicate more material stretching above a certain  $\lambda$  value. A more equal division of rotational and elongational flow produced more stretching. We will present these ideas in a complete and comprehensive way to share our understanding of the rheological basis of bubble formation in mixers, extruders and in foam-like cereal products.

## PLENARY 10.

### **Deformation, orientation and bursting of microcapsules in simple shear flow: Wrinkling processes, tumbling and swinging motions**

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Microcapsules with polymer membranes as wall materials are often synthesized for technical, cosmetic, medical and pharmaceutical applications on grounds of their well-defined controlled release properties. Due to their simple structure and viscoelastic properties these particles are also used as artificial model systems to mimic the mechanical properties of biological cells. The investigation of bursting processes, capsule deformation and orientation in linear shear flow is, therefore, of great interest for technical applications and basic scientific research.

In a series of experiments we investigated the rheological properties of polysiloxane and polyacrylamide microcapsules in an optical flow cell (rheoscope). In additional experiments we measured the regime of linear viscoelastic response and the shear rheological properties of the flat membranes in an interfacial rheometer. In spinning drop experiments, we determined the surface Young moduli and bursting process of the artificial particles. Measurements of the bending rigidity of the membranes were obtained from pendant-drop-experiments. The combination of these four experiments allowed evaluating the mechanical properties and the surface Poisson ratios of the capsule membranes.

In experiments of simple shear flow we noticed that the capsule deformation was influenced by many parameters such as the viscosity ratio between the inner and outer phase, the water concentration of the oil phase, the polymerization time and the membrane bending rigidity. Besides these phenomena we observed a large number of different rheological processes like shear induced membrane wrinkling, tumbling, swinging and other dynamic motions. In additional experiments we investigated the flow induced bursting process of the viscoelastic capsules. It turned out that most of the non-linear experimental results were in fairly good agreement with new theoretical approaches and recent simulations.

**Keywords:** *microcapsule, simple shear flow, shear induced deformation, membrane elasticity, capsule orientation, capsule bursting, tank-treading*

## PLENARY 11.

### Heat Transfer Asymptote in Laminar Tube Flow of Non-linear Viscoelastic Fluids

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The state of the knowledge in heat transfer with non-linear viscoelastic fluids flowing in straight tubes of *circular and non-circular cross-section* is reviewed. Recent findings related to the counterpart in *laminar flow in non-circular tubes* of the well-known Hartnett heat transfer *asymptote in turbulent flow* of non-linear viscoelastic fluids in *circular pipes* are reported.

The fully developed velocity and thermal fields in constant pressure gradient driven laminar flow of a class of non-linear viscoelastic fluids with instantaneous elasticity in straight tubes of arbitrary contour  $\partial D$  with constant wall heat flux is investigated to show the existence of a heat transfer asymptote in laminar flow of viscoelastic fluids in *non-circular* tubes. This asymptote is the counterpart of the heat transfer asymptote in turbulent flows in *circular* pipes of non-linear viscoelastic fluids. The asymptote delineates the boundary between the region where enhancement is a function of inertia alone and the region where it is a function of both inertia and elasticity. A different asymptote corresponds to different cross-sectional shapes in straight tubes. The asymptotic independence of the Nusselt number  $Nu = f(Re, Wi) \rightarrow Nu = f(Re)$  from elastic effects represented by the Weissenberg number  $Wi$  with increasing  $Wi$  is shown analytically in *non-circular* contours such as the ellipse, the square and the equilateral triangle. The average Nusselt number  $Nu$  is a function of the Reynolds  $Re$  and the Prandtl  $Pr$  numbers, represented by the Péclet number  $Pe$ , and the Weissenberg number  $Wi$ .

The class of non-linear fluids considered is constitutively represented by single mode, non-affine differential constitutive equations which include the Johnson-Segalman and the modified Phan-Thien-Tanner (MPTT) fluids as well as affine differential constitutive equations of the Giesekus type. The driving forces can be large. Asymptotic series in terms of the Weissenberg number  $Wi$  are employed to expand the field variables. A continuous one-to-one mapping is used to obtain arbitrary tube contours from a base tube contour  $\partial D_0$ . Heat transfer enhancement due to shear-thinning is identified together with the enhancement due to the inherent elasticity of the fluid. The latter is to a very large extent the result of secondary flows in the cross-section but there is a component due to first normal stress differences as well. Increasingly large enhancements are computed with increasing elasticity of the fluid as compared to its Newtonian counterpart. Order of magnitude larger enhancements are possible even with slightly viscoelastic fluids. The coupling between inertial and viscoelastic nonlinearities is crucial to enhancement. The change of type of the vorticity equation governs the trends in the behavior of  $Nu$  with increasing  $Wi$  and  $Re$ . Slight deviations from Newtonian behavior triggers a rapid rise in enhancement as opposed to the behavior for larger values of the Weissenberg number  $Wi$  where the rate of increase is much slower, and ultimately tends to level off asymptotically. The asymptotic independence of  $Nu$  from elasticity with increasing  $Wi$  is related to the extent of the supercritical region controlled by the interaction of the viscoelastic Mach number  $M$  and the Elasticity number  $E$ , which mitigates and ultimately cancels the effect of the increasingly strong secondary flows with increasing  $Wi$  to level off the enhancement.

## PLENARY 12.

### Statement and Resolution of Generalized Orr-Sommerfeld Equation

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The Orr-Sommerfeld equation governs the stability of a parallel flow in the x-direction for a Newtonian fluid submitted to 2D perturbations in the (x-y) plane. It writes [14; 19]:

$$\left( \partial_t + U \partial_x \right) \Delta \psi - \partial_{yy} \bar{U} \partial_x \psi = \frac{1}{\text{Re}} \Delta^2 \psi \quad (1)$$

Where  $t$  denotes the time,  $\psi$  the stream function,  $\text{Re}$  the Reynolds number and  $\bar{U}$  the average velocity. The Newtonian fluid can model a debris flow [2; 3; 9; 11]. A debris flow can also be modeled by a suspension (Nsom, 2001) and more generally by a Herschel-Bulkley fluid, characterized by viscoplasticity and shear-thinning properties [8]. At this point, some assumptions can be made to simplify the equations obtained ([5; 6]) and a phenomenological stability study can be brought out [1]. However, a more general approach can be formulated without resorting to such assumptions [18]. Unfortunately, the equations obtained are dissuasive. So, there is a need of a rigorous model for the stability of a debris flow.

Indeed, Ng and Mei [7] state that at a low shear rate, the power-law model which only takes into account the shear-thinning property of a mudflow [20; 4; 13] and neglects its plasticity is more appropriate. Perazzo and Gratton [16; 17] investigate general characteristics of gravity-driven shallow layer of a power-law liquid on an incline.

The rheological behaviour of the mud, modelled by its shear-thinning property, obeys the so-called power-law equation, which makes it possible to represent changes in viscosity as shear increases [15]:

$$\underline{\sigma} = 2\eta \sqrt{-\underline{D}} \quad \text{and} \quad \text{tr}(\underline{D}) = 0 \quad (2)$$

$$\underline{D} = \frac{\text{tr}(\underline{D}^2)}{2} \quad \text{and} \quad \eta = k(-\underline{D})^{(n-1)/2} \quad (3)$$

where  $\underline{\sigma}$  denotes the extra-stress tensor, the constants  $k$  (liquid consistency) and  $n$  (power-law index) are the rheological parameters, while the two scalars  $\underline{D}$  and  $\eta$  are the second invariant of  $\underline{D}$  and the apparent viscosity, respectively. Nsom et al. [12] computed and established new characteristics of a muddy debris flow generated by a dam breach in that model.

In this paper, we consider the stability of a power law fluid in a long domain, i.e. a flow domain with one dimension much greater than the two others, practically a flow of unloaded mud in an inclined open 1D channel. Applying the equations of conservation of the mass and the momentum, an equation of motion of the steady basic flow are derived and solved. Then, a 2D perturbation is superimposed to that solution and the equation of motion obtained is then linearized. It has the form of eq. (1), but with additional terms depending on the rheological parameters. Moreover, putting  $n = 1$  in that equation, eq. (1) is recovered, so we named it a generalized Orr-Sommerfeld equation. That equation was then solved numerically using a shooting method and analytically, to discuss the flow stability.

**Key words:** Muddy debris flow, Open channel, Orr-Sommerfeld equation, Power-law fluid, Slope, Stability

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## **PLENARY 13.**

### **Rheological Bases of Development of Materials with Extreme Dynamic Strength**

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It is obvious that materials with different chemical composition and material structures have different chemical, physical, mechanical and thermal properties. Some of these materials have excellent elasticities, meanwhile the others are rigid and susceptible to brittle fracture. Another group of substances are viscous and having a wide range of viscosity depending on temperatures and speeds of deformation or sheare ratio. Other materials are plastics and have different values of statical yield points. It is also clear that the different crystalline materials have different values of elasticity modules and different melting temperatures.

Having several years experiment in investigation of high-speed collisions and in research of rheological properties of ceramic matrix composites and hybrid materials the authors successfully understood the rheological principles of development hetero-modulus, hetero-viscous and hetero-plastic complex materials with extreme dynamic strength. Generally good dynamic strength can be achieved with material compositions prepared and made from components having different modules of elasticities from low values like carbon and light metals (Mg, Al, Ti) up to very high values like boride, nitride and carbide ceramics. Thanking to the local temperature increase and phase transformations in the materials during high-speed collisions the better dynamic strength can be reached when material compositions are built up from components having not only different elastic modules but different melting temperatures and thermal conductivities.

In their work the authors underpin that materials with better dynamic strength can be obtained when adding hetero-viscous and hetero-plastic particles and elements to hetero-modulus components.

**Keywords:** *collision, dynamic strength, elasticity, modulus, rheology, viscosity, yield point*



**SESSION 1**

**Applied Rheology and Modeling of Clays,  
Minerals and Rocks**

## **Seismic Based Rheological Parameters Provide Detailed Lithological and Structural Subsurface Images In Mining Exploration**

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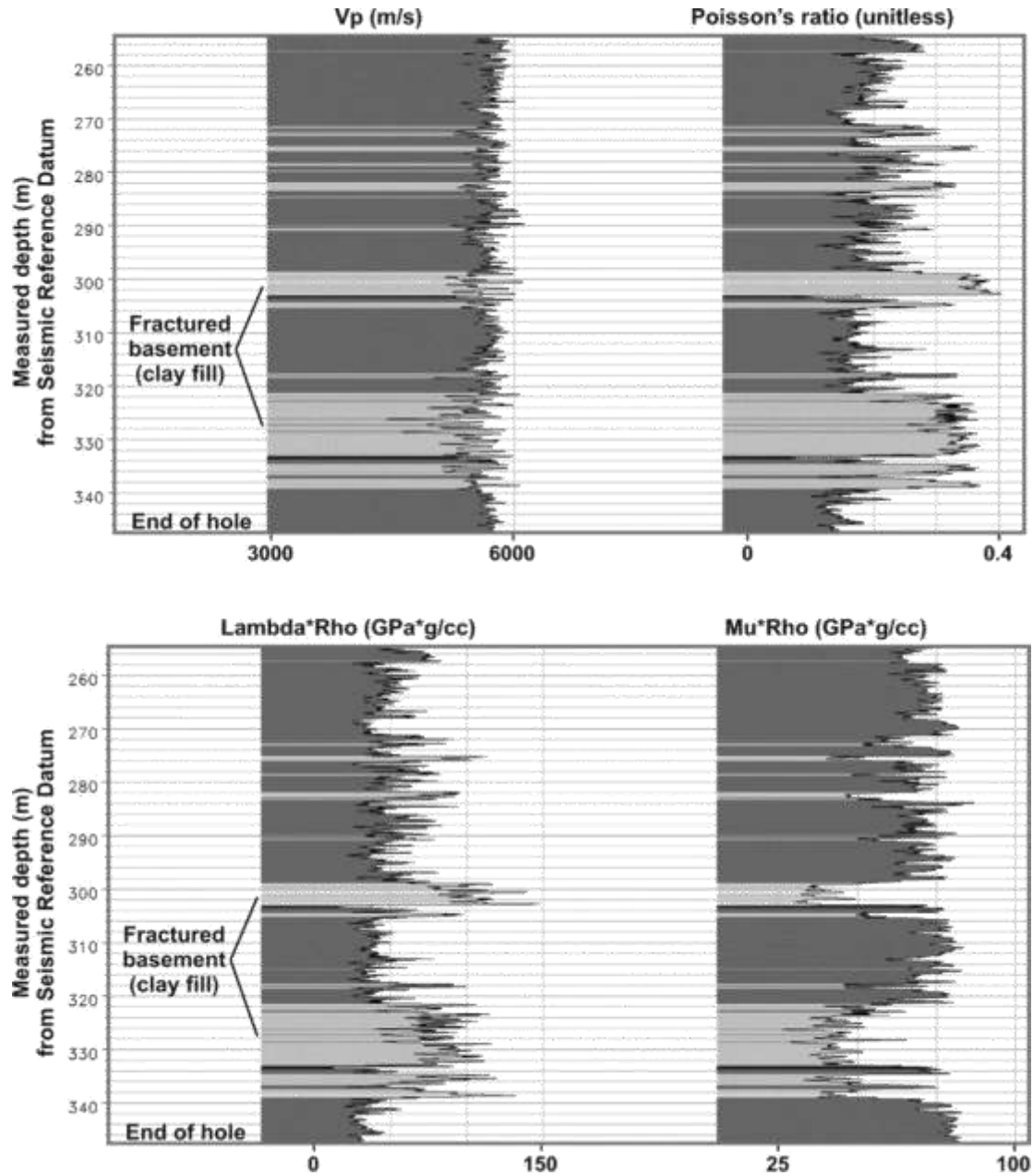
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The Meso-Proterozoic Athabasca Basin is recognized for its numerous high grade uranium mineralizations. Only the shallow margins of this 100,000 km<sup>2</sup> depression have been effectively explored up to this date. With intent to extend the activities into the deeper part of the basin, seismic reflection surveys were introduced in the early 2000s. Seismic images effectively recognize deep basement faults/shear zones, the unconformity (UC) between the sandstone basin fill and the crystalline basement. It is also well recognized (Goodway, 2001; Ikelle and Amundsen, 2005; Russell & Hampson, 2006) that the seismic shear waves (S) and compressional waves (P) are controlled by different rheologic parameters, thus extraction of these elastic parameters from the seismic signal may be utilizable to detect lithologies, their variations, characterization of permeable zones (fractures), and mineralization-related clay alteration halos. This study is our first attempt in the basin to extend this combined seismic and borehole data analysis with intention to determine the effectiveness of these intrinsic signal properties as indicators of favourable mineralization target zones. Five Full Wave Sonic logs and two Optical Televiewer observations were utilized for this investigation.

Density-depth values were established through known compressional wave velocity ( $V_P$ ) and density ( $\rho$ ) relationship. Specific lithology/alteration related parameters, acoustic impedance ( $V_P$ ),  $V_P/V_S$  ratio, Poisson's ratio, Lambda-Rho ( $\lambda\rho$ ) and Mu-Rho ( $\mu\rho$ ) were computed for every depth point of the boreholes. Recent investigations (Goodway, 2001) demonstrated that interconnected relationships of these rheology parameters provide more demonstrative indications of distinct lithologies, alteration zones or anomalous fracture zones than their single values alone. Cross-plots of these combined parameters help to recognize several characteristic hierarchical clusters associated with distinct fracture zones within specific lithologic entities and prominent alteration intervals. High velocity related clusters are a consequence of silicification of the sandstone; low velocity intervals are direct indications of clay alteration (**Figure 1**).

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**Figure 1:** Parameter logs derived from Full Wave Sonic (FWS) survey showing the elastic properties of clay filled fracture zones in the crystalline basement of the Athabasca Basin. For the investigated (clay) zones compressional wave velocity ( $V_p$ ) is low, Poisson's ratio is high,  $\Lambda \cdot \rho$  is high, and  $\mu \cdot \rho$  is low ( $\rho$  is the density of the rocks). This separation of the elastic parameters provides a useful tool to detect clay alteration zones from 1-D FWS measurements and also from 2-D or 3-D surface seismic observations.

**Keywords:** Full Wave Sonic logging, elastic parameters, Poisson's ratio,  $\Lambda$ ,  $\mu$ , cross-plot analysis, Athabasca Basin, crystalline basement, clay, alteration, uranium mineralization.

## **Rheological properties and stability of Latvian illite clay-glycerol suspensions**

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Clays and clay minerals are widely used in cosmetics, health care and therapeutic products as excipients and active ingredients. The most important properties for these applications are high specific area and adsorptive capacity, chemical inertness and rheological properties. Most cosmetic and health care products are designed for topical applications, which require appropriate rheological properties. These products must flow easily when deformed and retain their shape in repose. Solid-in-liquid and liquid-in-liquid systems in cosmetic products are thermodynamically unstable and the phases tend to separate with time. The addition of clay minerals improves the stability and rheological properties of suspensions and emulsions, therefore they are used as excipients and thickening agents in many semi-solid preparations.

An important factor for use of clay minerals in cosmetics and health care is the purity. Most naturally occurring clays typically are mixtures of clay and non-clay minerals. The value of clays depends on the required purification, which accordingly depends on the type and amount of non-clay minerals. In Latvia the most abundant clay mineral is illite, but the clays can also contain kaolinite and non-clay minerals, such as quartz, feldspar, calcite, dolomite, metal oxides and hydroxides and muscovite. Latvia has large clay reserves, but they are traditionally used in building materials and pottery. To use Latvian clays as excipients in cosmetic and therapeutic products, it is necessary to perform simple, but effective purification process and to investigate the rheological properties of purified illite. In this study, the rheological properties and stability of illite-glycerol suspensions were investigated. Glycerol is often used in semi-solid cosmetic and health care products due to its moisturizing, soothing and healing properties.

Illitic clays from two deposits in Latvia were used. Carbonates were removed by dissolution in two different acids - citric acid and hydrochloric acid. Gravity sedimentation was used to remove quartz and feldspar and to obtain clay fraction < 2 µm. The XRD data shows that the obtained clay fraction contains mostly illite and also small amounts of kaolinite, quartz and feldspar. The rheological measurements were taken at room temperature 25±1°C and at shear rate 1-200 rpm. The stability of clay suspensions was evaluated using zeta potential measurements.

**Keywords:** *rheology, illite, glycerol suspensions, stability, zeta potential*

## **Rheological investigations of tailings of kimberlite ore dressing and numerical simulation of its behavior in PLAXIS**

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The article presents the results of analysis of rheology properties of sand-clay tailings (wastes) of kimberlite ore dressing of the diamond deposit (Russia, Arkhangelsk region). The sand-clay tailings are represented by coarse, medium, fine and silty sands with the content of clay particles up to 20%. The tailings have a specific deformation and strength characteristics. The tailings are foundation bed for each tier of the 3-tiered dam erected at the diamond deposit and have a influence on dam's safety in terms of long-term consolidation process of the sand-clay tailings.

The coefficient of secondary compression as main parameter of soil's creep is defined by implementing standard one-dimensional consolidation test. The sample had the following dimensions: diameter - 73 mm, height - 20.85 mm. Tests were carried out under vertical pressure: 0.1, 0.2, 0.3 MPa. Coefficient of secondary compression was defined for different initial void ratios of the samples. We obtained the linear correlation between initial void ratio and coefficient of secondary compression of sand-clay tailings.

For numerical simulation of behavior of sand-clay tailings subject to its rheology properties in PLAXIS software we used Soft Soil Creep (SSC) model (time independent behavior). The main parameters of the model are: compression index ( $C_c$ ), recompression index ( $C_r$ ) and coefficient of secondary compression ( $C_a$ ).

To assess the accuracy of the SSC model we carried out numerical simulation of standard onedimensional consolidation tests. We used axially symmetric geometry model for simulation the consolidation cell. We modelled all loading/unloading phases as we made during compression test. Results of numerical simulation and consolidation tests have a good correlation.

The obtained rheological parameters of sand-clay tailings let us perform the prediction of the behavior 3-tiered dam for a long time on basic of results of numerical simulation in PLAXIS.

**Keywords:** *sand-clay tailings, consolidation test, coefficient of secondary compression, numerical simulation, Soft Soil Creep model, PLAXIS*

## **New Approach for Development of Rheological Relations for Saturated Porous Media**

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Hydro-geo-mechanical models are the basis of the solution of important problems in hydrogeology, hydro-geo-ecology, oil production, geophysics. Oil depletion on some of the Russian deposits necessitates formulating models that take into account the variation of deflected mode caused by chemical interactions between components of underground fluid and the material of the porous skeleton to perform effective enhanced oil recovery. Those models are also essential in problems of hydrogeology, such as filtration of solutions in clay layers, suffosion processes and karst processes. Usually, chemical interactions, which were mentioned above, cause variation of the mass of the porous matrix. That is why it is important to perform an additional research on the influence of this variation on rheological relations, which are required to obtain closure model of deformations of a filtrating porous medium. It is also necessary to perform systematic development of the main equations of the underground mass-transfer in this case. Whereas those questions did not receive exhaustive explanation in specialized literature, it makes sense to obtain required equations and examine most important applications. Equations of hydro-geo-mechanics in filtrating porous media with porous variable-mass skeleton are examined. Variation of the skeleton's mass occurs due to heterogeneous chemical reactions. Regularities of mass transfer and deformations in saturated porous media are analyzed. Peculiarities of obtaining of rheological relations are investigated.

**Keywords:** *rheology of saturated porous media, rheological relations, hydro-geo-mechanical model*

## **Rheological properties of the drilling muds.**

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Drilling muds in the process of drilling wells prevent possible technological complications, reduce layer pollution by using components of drilling mud, ensure the complete removal of drill cutting and provide the highest of the techno – economic indicators. Significant role in addressing these issues is hydraulic flushing drilling wells program, which is implemented by controlling the rheology of drilling muds. The main factor determining the effectiveness of the removal of drill cutting, is the ratio of plastic viscosity drilling mud with a dynamic strength to shear in the flow process. Behavior of the modern drilling muds processed by polymers with a high molecular weight, is believed to be described more precisely by the sedate law (Ostvald's – de Vaale model), rather Shvedov's – Bingham equation. This model has two rheological parameters:  $K$  – a consistence indicator and  $N$  – the indicator of nonlinearity characterizing degree of a deviation of rheological behavior of liquid from Newtonian. Unfortunately, the proper ways has not yet been developed to manage the nonlinearity of drilling muds. It is due to the fact that the study of the properties of drilling muds and their analytical description in the motion is carried out with the position of classical continuum mechanics. This does not take into account that the drilling mud is a continuous media at rest, whose properties are determined by the properties of bonds in it. We suggest to consider drilling mud as the discrete system consisting of the discrete dispersive media and a disperse phase. Thus it is necessary to consider that in drilling mud on an interface between two phases of disperse system there is a double electric layer which charges on the separate centers is presented discrete ions. The indicator of nonlinearity  $N$  will depend on ability of hydrogen bonds to form new bonds in mud and is an indicator estimating its internal continuous, and a consistence indicator  $K$  – the value of durability of hydrogen bonds. According to it, for pseudo-plastic liquid it will be fair that the more value  $N$  the more discrete the fluid is, and the more  $K$ , the bigger durability hydrogen bonds in volume of fluid possess is.

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## **Modeling of mechanical properties of materials obtained via alkaline activation of illite-based clays of Latvia**

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Alkaline activation is a chemical process in which a powder material of an aluminosilicate nature, such as clay, is mixed with an alkaline activator to produce a paste that is able to set and harden in short time. These materials, frequently termed alkaline inorganic polymers or geopolymers, constitute a new family of products which among other interesting properties can combine qualities typical of cements with those of traditional ceramics and zeolites.

Latvian clayey sediments were studied from the point of view of the national economy, analysing the physical properties and chemical composition of those sediments in connection with the possibilities of their utilisation in the manufacture of geopolymer materials. Clay generally consists of a mixture of different clay minerals and associated ones. The used Quaternary clay minerals dominate by illite (75-80%) with admixture of kaolinite and are typically rich in fine, scattered carbonates. The quantification of the clay mineral mixtures is difficult and needs experience and a combination of methods. The variability of clay composition and parameters of the chemical and thermal activation process complicate a general statement about the suitability of clay resources for the production of geopolymer materials.

Compressive strength and ceramic properties were determined and compared using different curing and thermal treatment conditions. Compressive strength measurements are widely used as an indicator to assess the success of inorganic polymer technology. This is due to the low cost, simplicity as well as due to the fact that strength development is a primary measure of the suitability of clays in various applications, int.al. for obtaining of geopolymers.

The aim of this study was to synthesize geopolymer products from illite clays of Latvia under alkaline activation. Results of the investigations showed the influence of alkalis on the transformation of clay/illite structure by curing of activated clays at different temperatures for various time. Obtained products were investigated by IR-spectroscopy and XRD, pore size distribution was determined as well. IR-spectra showed the differences among absorption frequencies for the untreated clays and activated ones and are a good indication of the transformations taking place during synthesis of geopolymer materials. It was stated that factors such as the presence of  $K_2O/Na_2O$  and  $CaO$  in the original clay, the type of alkali used in activation process and curing/thermal treatment had a significant correlation with the obtained compressive strength. Obtained results showed that illite-based clays of Latvia could be a source material for geopolymer production.

**Keywords:** *illite-based clays, alkaline activation, compressive strength*



## **Influence of grinding times on rheological and compacting properties of alumina and calcium-phosphate powder mixtures**

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The purpose of this research was to identify the effects of oxidic composition and grinding time on rheological, structural and compacting properties of high purity alumina and calcium-phosphate powder mixtures. The study involves the investigation of compacting properties of these composite powder mixtures depending on the milling time, compacting pressure, the appearance of amorphous phases and specific surfaces. To prepare specimens the atomiser powder of alumina-oxide was milled and mixed with the calcium-phosphate as additive in planetary ball mill. During the experiments the volume of calcium-phosphate additives were added from 0 m% up to 20 m% by 2,5 m%. The such prepared powder mixtures were compacted in uniaxial hydraulical press. On the basis of specific compaction – forming pressure stress and pressure - time curves the authors could successfully describe the changes of rheological behaviour and rheological model of the above powder mixtures. Increasing the compression pressure changes in the chemical composition and the phase transformations also could be observed in the alumina - calcium phosphate powder mixtures.

**Keywords:** *mechanochemical effects, rheological properties, calcium-phosphate, alumina powder, nano chemical effects, bioceramics*

## **Rheological Properties of Clay-based Compounds**

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The production of the traditional fired brick product demands the preparation and formation of the largest volume of plastic ceramic pastes. Additive materials are often mixed into the clay based compounds in order to change the behaviour of the clay or to improve the properties of the fired products such as thermal insulation. Effects of organic and non-organic additives on the rheological behaviour of clay-based compound were investigated in this study. As pore forming additives saw dust and ground sunflower seed shell, while as opening agent sand and ground brick dust were mixed to the clay matrix in 0, 3, and 5wt%. The water content value was set to 20% and 24% of the compounds. The investigation was performed by capillary rheometer. Results showed that the clay and as well as the compounds behaved as Bingham fluid in the measured interval. At 20% water content the pore forming additives and the ground brick dust increased the Bingham viscosity while the high purity quartz sand decreased it. The capillary flow showed mainly plug flow with extensive wall slip. By the help of sedimentated ( $d < 2\mu\text{m}$ ) clay minerals content of the clay, the slip layer was characterised by the layer thickness which was calculated from the data of measurements with capillary and rotational viscosimeter. The slip layer thickness showed a tendency to reach a limit value by increasing the extrusion speed.

**Keywords:** *brick, additives, Bingham flow, wall slip*

## **Session 2.**

# **Applied Rheology of Polymer Melts**

## **Rheological properties of wood polymer composites and their role in extrusion**

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Wood plastic composites (WPC) are an important and growing part of the plastic industry. In recent years WPC have evolved the considerable possibilities of design and functionality of the end product. The most common method for WPC a product manufacturing is either profile or sheet extrusion. The flow behaviour of the molten wood plastic composites is one of the critical parameters in the formation of the end product.

A better understanding of the rheological behaviour of the WPC and precisely measured material data are the pre-requisites for the process optimization and the accuracy of simulation of extrusion of a WPC. The flow properties of WPCs are strongly dependent on the wood filler content and on the moisture content of the wood particles. The moisture can have similar effect like a slip agent.

The main objective of this work was to analyse the flow behaviour of the PP based wood plastic composites, using a high pressure capillary rheometer with different melt pressure measurement techniques and dies with different geometry. The rheological experiments were performed using a slit and round dies. The measured viscosity data have been used in flow simulation of an extrusion profile die. Furthermore, the influences of different rheological models on the simulation results are demonstrated.

**Keywords:** *Wood Plastic Composites, Rheology, Slit Die, Wall Slippage, Extrusion Profile Die*

## **Numerical Simulation of Axial Crushes of Multiple Corners Square Composites Cotton Fiber Reinforced Propylene**

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In this paper the collapse behaviors of composite cotton fiber reinforced propylene were developed and analyzed, respectively. The crush studies with various regular polygonal cross-sections are numerically investigated under axial compression using the finite element method. The effects of wall thickness and velocity rate of impact on the crush behavior are also investigated. Crush strength increases as the number of corners of the cross-section increases, though it almost saturates for the number of corners beyond 10. Cross-sectional shape with less than five corners should be avoided to take a regulated collapse pattern. The effect of number of polygonal corners on enhancement in crush strength becomes more prominent as the initial wall thickness decreases. The validation finite element modeling was carried out with experimental analysis technique to ensure that the data results from numerical analysis will sufficiently accuracies.

**Keywords:** *Composite Structure, Finite Element Simulation, Axial Crushing, MALAYSIA.*

## **Investigate of the possibility of TEKCAST method application for Polymeric Materials Casting**

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The contribution gives an overview of knowledge about the method of centrifugal casting, with a focus on system Tekcast. Company Tekcast Industries has developed devices for centrifugal casting, which extend the area of production of prototype castings from metal or plastic. Materials suitable for the centrifugal casting with flexible operating parameters are uncoloured non-ferrous alloys or non-metallic materials such as polyester resins, polyurethane resins, epoxy resins, waxes and below. The casting process is suitable for a wide range of functional castings and decorative items.

**Keywords:** *Tekcast Method, technology of casting, polymers, mechanical properties, casting properties, centrifugal casting*

## **Elastic Recovery Lithography: Pattern Height on Demand using Stress Relaxation**

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With the proposed technique, one can create patterns with different feature heights using a single stamp on thin elastomeric films. This technique can generate imprinted patterns with lateral dimension identical to that of the stamp but having controlled feature height ( $h$ ) between 0 and  $h_0$ , where  $h_0$  is the height of the feature on the stamp. Creating patterns with different  $h$  would require multiple numbers of stamps in any existing soft lithography method. However, the same can be accomplished with a single stamp which is the key novel feature of this work. The technique is based on elastic recovery of visco-elastic Sylgard 184 films (a commercial thermocurable silicone based elastomer). Thermal pre-curing of as-coated Sylgard 184 films is done for different duration of time which results in partially cross linked films having different levels of visco elasticity. Subsequently, the same stamp under the same external force is used for imprinting the film. As the film is in a visco-elastic state, part of the energy corresponding to the deformation is stored within the flexible cross linked matrix of the film (component EE) and part of it is dissipated due to viscous dissipation (component EV). The relative proportions of EE and EV depend on the film rheology, i.e. on storage modulus ( $G'$ ) and the loss modulus ( $G''$ ), which in turn are functions of duration of pre-curing. Once the external load is withdrawn, the energy stored in the form of elastic deformation within the film matrix (EE) relaxes, eventually trying to flatten the film surface. Thus, pre cured films with different extents of reduction in the amplitude of the imprinted patterns results in features with different  $h$ . Superhydrophobic surfaces with high water contact angle ( $WCA > 150^\circ$ ) are fabricated using the biomimetic route. The process of replica molding is used to create double replicated positive replicas of top surface of a Lotus leaf. This surface exhibits a Cassie state of wetting ( $WCA = 150^\circ$ ). Using the principle of Elastic Recovery, positive replicas of Lotus leaf can now be created with pre-curing. These surfaces exhibit Wenzel state of wetting with lower water contact angles. Hence the technique can also be used to study how the geometry of the patterns influence the wettability of the surfaces and fabricate surfaces with gradient wetting regimes.

**Keywords:** *Soft Lithography, Viscoelasticity, Stress Relaxation, Viscous dissipation, Wettability*

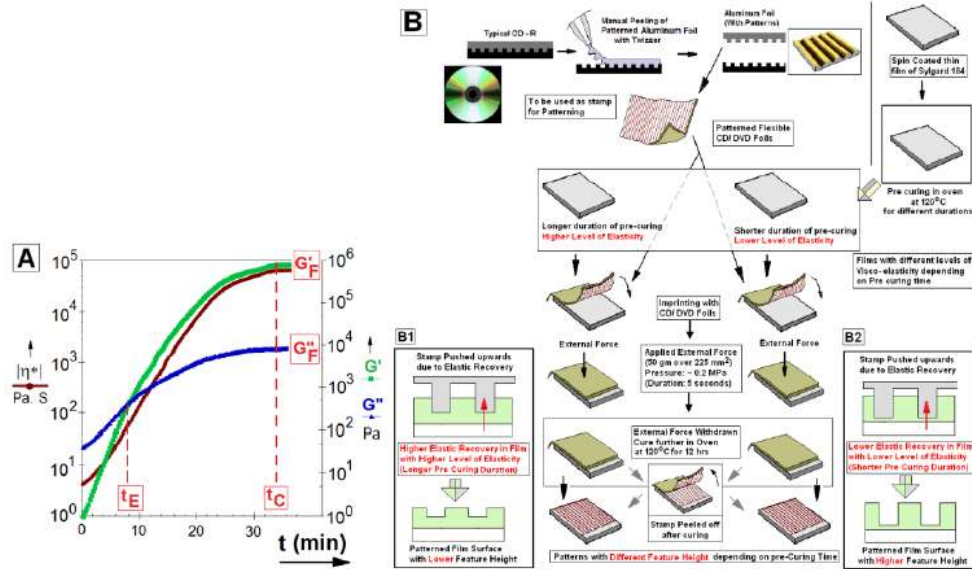


Fig: (A) Rheometer data indicating the progressive change of  $G'$ ,  $G''$  and  $\eta^*$  with time, during the early stages of cross linking of PDMS. (B) Schematic illustration of the mechanism leading to the formation of imprinted patterns with different feature height using the same stamp on Sylgard 184 film surface.

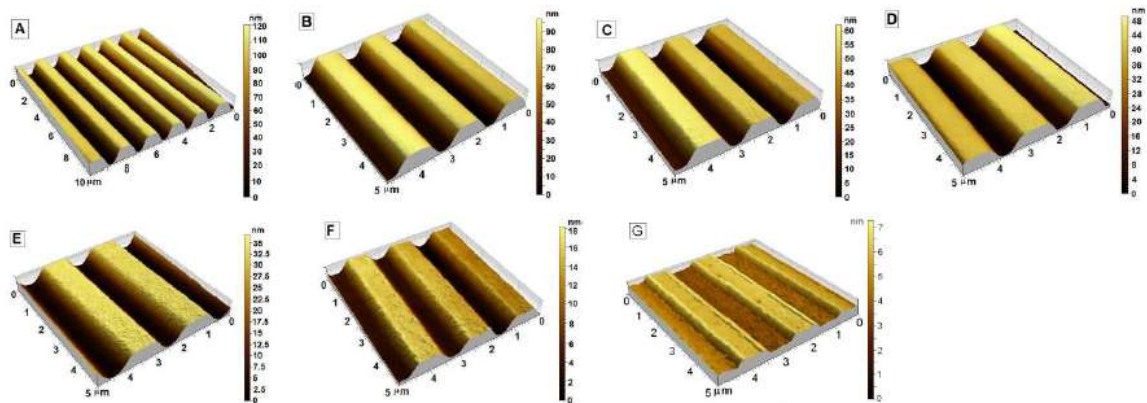


Fig: Structures with different feature height formed due to different levels of precuring before imprinting. Stripe height (A) 120 nm (no precuring); (B) 95 nm (5 min pre-curing); (C) 63 nm (10 min pre-curing); (D) 50 nm (15 min pre-curing); (E) 37 nm (20 min pre-curing); (F) 22 nm (24 min pre-curing) and (G) 7 nm (27 minute pre-curing). After 30 minutes of precuring no pattern was seen on the film surface. In all cases, a CD foil was used as the stamp.



## **Correlation between Rheotens Measurements and Reinforcement of Polymer Nanocomposites in the Injection Molding Compounder**

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Usually the reinforcement of polymers and polymer nanocomposites, in particular the improvement of Young's modulus were performed using tensile tests. Structural and morphological characterizations were normally investigated using expensive techniques like transmission electron microscopy (TEM), X-ray scattering and sometimes also rheological analyses (rotational rheometry). The objective of this study is to generate faster and cheaper data to verify the quality of the produced compound in an On-Line measurement system and subsequent processing of injection molded parts "by only one plasticizing process" by the use of the Injection Molding Compounder (PNC-IMC). In comparison to the conventional compounding process, where the compound has to be pelletized and fed into the injection molding machine for the second plasticizing process, the injection molding compounding combines these two processing steps.

This paper shows the first results and problems with the implementation of the rheotens equipment into the concept of the Injection Molding Compounder. Different processing techniques and various processing conditions were compared and the occurring effects were tried to detect both with tensile testing and extensional melt rheology. The increase of the Young's modulus by using nanofillers compared to the virgin polypropylene and the correlations of the level of melt strength with these values is shown.

These very first results give a good overview on both the possibilities and the limitations of the material pre-tests by the use of extensional rheology in the concept of the PNC-IMC. Further studies to enable a fast and efficient way of estimating the level of reinforcement in polymer nanocomposites by the use of rheotens measurements will be done in order to provide usability for the industry also. Measurements with the small angle X-ray scattering method are also planned to verify the exfoliation and intercalation of the layered silicates in the polymer matrix.

**Keywords:** *Injection molding compounding, Polymer Nanocomposites, Extensional Rheology, Rheotens*

## **Rheological Characterization of Polyoxyethylene (POE) and Carboxymethyl Cellulose (CMC) Suspensions with Added Solids**

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Knowledge of the rheological behavior of polymeric fluids is fundamental to understand the processing behavior and relations between structure and property of these fluids. In various industrial fields, water-soluble polymers are commonly used as thickening agents to control the rheology of aqueous fluids. Polyoxyethylene (POE) is a polymer of ethylene oxide with many applications from industrial manufacturing to medicine; liquid and waxy polymers with low molecular weights are widely used in formulating pharmaceuticals, cosmetics, pigment dispersants, spreading agents in agricultural sprays, etc; applications of high molecular weights polymers depend on their solubility, rheology and thermoplastic behavior. Carboxymethyl cellulose (CMC) is derived from cellulose, it is used as a viscosity modifier or thickener, and to stabilize emulsions in various products including ice cream; it is known for its excellent water holding capacity.

The aim of this research was to study the changes in apparent viscosity of high molecular weight POE and CMC suspensions by adding micro-metric solid particles such as fibers or spheres.

POE concentration varied from 1% to 1.5% w/w and CMC varied from 1.5% to 2.2% w/w. Spherical solids varied from 0 to 50  $\mu\text{m}$  and from 40 to 70  $\mu\text{m}$ ; cylindrical solid particles varied from a length of 2-3 mm and a length/diameter ratio of 11 and 21. Solid particle concentrations varied from 10%; 20%; 30% and 40% for each range and geometry.

A rheometer (Carri Med, CSL2 100, TA Instruments, England) using a 40 mm diameter parallel plate fixture was used to carry out the experiments. The lower plate is equipped with a Peltier temperature control system; all tests were conducted at 20°C with 3 repetitions. Results were analyzed and different rheological models were adjusted with good accuracy (Carreau and Power law models among others). In particular, it was found that the solid particles addition decreased apparent viscosity for all the samples. As suspension concentration increases apparent viscosity decreases.

**Keywords:** *Rheology, Polyoxyethylene, Carboxymethyl cellulose, Power law, Carreau*

## **Mathematical characterization of the values of rheologic variables during the networking reaction of rubber mixtures based on SBR**

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The article presents an analysis of crossbridging reaction using measurements of time dependencies of torque at constant temperature from the range 100 up to 200 ° C. The measured results obtained on sample based on styrene - butadiene (SBS) prepared in the laboratory show the behaviour which can be well described by equations of chemical reactions of first-order kinetics. It is possible mathematically describe significant constants of the kinetics of networking reaction (induction period, reaction rate coefficient) by the solution of differential equations and by mathematical approximation. Constants are exponentially dependent on the temperature of vulcanization, while dependencies are Arrhenius like. Math description allows describe the progress of the vulcanization reaction also in the temperature range outside of the monitoring interval, i.e. in the area of extremely long times required for the realization of the crossbridging reaction.

This research was supported by APVV-SK-CZ-0168-11, VEGA 1/0356/13 and ITMS:26220120014

**Keywords:** *vulcanization reaction, torque, mechanical properties, styrenebutadiene rubber*

## Monitoring of vulcanization process using measurement of electrical properties during linear increasing temperature

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The article presents the possibilities of diagnostics of irreversible chemical reaction - vulcanization in case of laboratory prepared rubber mixture based on styrene - butadiene (SBR) using measurements of selected physical parameters. Our work is focused on the measurement of current rheologic parameters (torque at defined shear deformation) and selected electrical parameters (DC conductivity) during linear increasing temperature. The individual steps of vulcanization are well identified by means of measurements of rheologic parameters, while significantly affecting the value of the electrical conductivity. The value of the electrical conductivity increases with the increasing of rate of the crossbridging reactions during vulcanization (fig. 1). The rate of the heating affects both types of measurements. When the rate of the heating is increasing the temperature of the beginning of networking step of reactions and also the rate of vulcanization grow. The sensitivity of the both types of measurements allows a good mathematical description of the temperature dependence of the torque and the electric conductivity during the vulcanization of rubber mixtures based on SBR.

This research was supported by APVV-SK-CZ-0168-11, VEGA 1/0356/13 and ITMS:26220120014

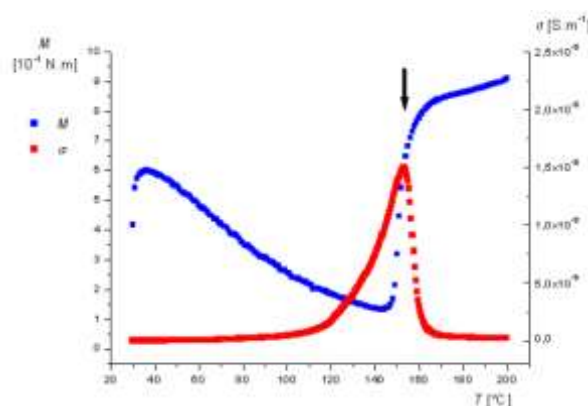


Fig. 1 Correlation between electrical and rheological measurements with linearly increasing temperature

**Keywords:** vulcanization reaction, electrical conductivity, torque, mechanical properties, electrical properties

**Session 3.**  
**Applied Rheology  
and Material Structure of Foods**

## **The rheological properties of Psyllium gum in protein based solution**

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In this study the rheological behavior of Milk Protein Concentration (MPC)/Psyllium gum mixture was investigated in aqueous solution at Pasteurized and non Pasteurized state. Psyllium gum was extracted and dried at 50°C for 48h. It was added to MPC solution in five concentration levels (0,0%0.01%,0.03%,0.06%,0.09%). In all treatments the MPC concentration was kept at a constant level of 1.5%w/v. Rheological analysis was performed at 25°C. The Results of the non-pasteurized state revealed that in treatments(0,0%,0.01%,0.03 %,0.09%), solutions exhibited shear thickening behavior while at levels (0.06%), at the first, shear thinning behavior was observed then at the higher shear stress, solution showed shear thickening behavior. The pasteurized state revealed that in these gums concentrations (0.01%,0.06%,0.09%), solutions had shear thickening behaviors while at levels control (0.0%), shear thinning behavior was observed then in higher Shear rate shear thickening behaviors while at level (0.03%), Newtonian fluid behavior observed in lower shear rates but it changed to shear thickening in the higher rate.

## **Rheological modeling of olive oil/policosanols organogels**

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Fatty alcohols are already known for their potential application as organogelators for organic solvents (oils). In particular, policosanols are a mixture of fatty alcohols often used for nutraceutical aims, being recognised, for example, as an enhancer for athletic performances. In the present paper, the use of such food-grade additive as vegetable oils organogelator has been investigated by preparing samples based on virgin olive oil at different policanol concentrations, in order to perform a rheological optimisation aimed at design new products for different food applications. For each sample, dynamic temperature ramp tests were carried out, and their analysis allowed the onset of crystallisation temperature ( $T_{co}$ ) to be evaluated and the gelation temperature ( $T_g$ ). Moreover, the rheological characteristics of samples at temperatures lower than the crystallisation onset were studied with frequency sweep tests at 25°C.

Experimental results evidence that the lowest policanol concentration at which crystallisation takes place is 0.1% w/w, and crystals and their aggregates start to interconnect yielding the samples gelation (i.e. the formation of a structured 3D network) for concentrations larger than a critical value ranging between 0.3% and 0.5% w/w.  $T_{co}$  increases nonlinearly with organogelator fraction, and an asymptotic trend seems to be reached for concentrations larger than 50% w/w. A fractal model was used to fit experimental storage modulus data,  $G'$ , as a function of policanol fraction, and the resulting fractal dimension was used as a fitting parameter in a phenomenological equation based on a modified fractal model, and proposed to fit experimental  $T_{co}$  values as a function of the policanol fraction.

**Keywords:** *policanol, olive oil, organogel, rheology, crystallisation*

## **An olive oil/policosanol organogel for Ferulic Acid controlled delivery**

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Crystalline organogels are structured semisolid systems, whose rheological characteristics are due to the assembly of low molecular weight organogelator molecules (LMWOs) which crystallises under particular process conditions. Thus, the inner microstructure of organogels is based on a 3-D network, into which both an organic liquid solvent and other dispersed particles can be entrapped. In the present work, olive oil organogels were structured with fatty alcohols (policosanol) and a rheological characterisation was carried out to find the best formulation for producing a support for Ferulic Acid delivery *via* oral administration. Ferulic Acid is a nutraceutical compound known for its antioxidant properties.

The rheological optimisation of the olive oil-policosanol organogels was based on Step Shear Rate Temperature Ramp tests, cooling samples from temperatures higher than the onset of crystallisation down to cold conditions, according to the samples crystallisation characteristics.

It was found that a policosanol weight fraction of 0.03 was enough to obtain an organogel having properties suitable for the desired applications, being contemporary semisolid, consistent enough and thermally stable for human ingestion. *In-vitro* tests on organogel loaded with Ferulic Acid were furthermore carried out in order to simulate the oral intake of the nutraceutical compound, evidencing a good performance of gel, which was found to delay and control the bioavailability of the molecules during the digestion processes.

**Keywords:** *policosanol, olive oil, Ferulic Acid, controlled delivery, organogel, rheology*



## **Effect of different hydrocolloids on rheological properties of model system jam: Simplex Lattice Mixture Design Approach to optimize the hydrocolloid levels**

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In the current study, different hydrocolloids (xanthan gum, carboxymethyl cellulose (CMC) and pectin) were used for the preparation of the model system jam in addition to sugar and citric acid and rheological characteristics were determined using a controlled stress/strain rheometer at a constant temperature. Different model system jam mixtures were prepared according to simplex lattice mixture design to determine the interaction between the hydrocolloids and optimize the hydrocolloid levels according to the desired properties of model system jam. Steady shear (consistency coefficient and flow behavior index) and dynamic shear (loss modulus, storage modulus, complex viscosity, complex modulus and tangent delta) rheological properties were studied and these parameters were modeled to construct some regression models and optimize their levels for a high quality jam production. Consistency coefficient, flow behavior index and apparent viscosity values of model system jam samples were determined to be in the range of 0.1616-1.1362 Pa sn, 0.6391-0.9826 and 0.1292-0.3806 Pa s, respectively. Simplex lattice mixture design application showed that the processing levels can be easily optimized depending on the requested rheological parameters. The results concluded that the coefficients of determination for the studied parameters was determined to be higher than 0.7153. The linear effects of used hydrocolloids on the rheological properties of jam was determined to be significant statistically ( $p < 0.01$ ). Each hydrocolloid showed an increasing effect on the all rheological parameters. The optimization results showed that CMC among the hydrocolloids should be used at 100% proportion definitely in the model system jam to provide a highest increase in the consistency coefficient of jam. The optimization results of this study can be used to manufacture a high quality jam.

**Keywords:** *model system jam, hydrocolloid, rheology, mixture design, optimization*

## **Effects of Ripening on Rheological Properties of Avocado Pulp (*Persea americana* mill. cv. Hass)**

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Avocado (*Persea americana* Mill), an American origin fruit tree whose fruits were known and consumed by natives of Mexico, Central and Northern South America, was introduced in Peru and Chile during the Inca Empire. In Chile, Hass is the most planted variety and with a greater trade prospect. Hass peel is thin, smooth and soft, intense glossy green. The pulp has fiber and its oil content varies between 18 and 22 % reaching values of 30%. Avocado composition depends on variety, geographical area of growth, maturity, soil characteristics and environmental conditions. The aim of this study was to investigate the effect of maturity on rheological properties of climacteric Chilean Avocado Hass fruit (pulp) (*Persea americana* mill. cv. Hass). Fresh unripe avocados (*Persea americana* mill. cv. Hass) were obtained from INIA-La Cruz, Valparaíso- Chile. They were washed and hand peeled. Pulp was placed in a cutter and mixed with 1.5% (w/w) sodium chloride and a citric acid: ascorbic acid ratio (3:1) to reduce the initial pH to 4.1. Approximately 150 g of avocado puree were poured into plastic whirl-pack bags, heat sealed and maintained under refrigeration no longer than 3 h until rheological tests were performed. Flow curves were obtained for the avocado puree using a rheometer (Physica-Rheolab MC120/UM, Physica U.S.A., Inc., Spring, TX). Avocado puree temperature was controlled at 20°C. Tests were carried out using a cone and plate geometry with 0.8-mm gap. Six intervals spanning from 0.1 to 100/s, increasing and decreasing in a linear ramp, were performed on each avocado puree. Data was plotted and analyzed for different rheological models. Viscoelastic properties of avocado purees were analyzed using small amplitude oscillatory shear. Avocado puree temperature was kept at 20 ± 0.1°C. Data was collected in the linear viscoelastic region and reported as  $G'$  (storage modulus) and  $G''$  (loss modulus). Analyses were repeated to provide the mean of seven determinations for each data point. Rheological data were adjusted to power law and Herschel-Bulkley models to determine rheological properties. Results showed that for fresh and aged avocado pulp a Herschel-Bulkley model best represents its behavior.

## **Rheological parameters' of dough with inulin addition and effect on bread quality**

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The goal of this study is the rheological evaluation of enriched flour prepared with an addition of inulin as a source of biologically active components in nutrition. Inulin is a storage carbohydrate found in many various plants like chicory and Jerusalem artichoke. It contains fructan chains of different degree of polymerization. It is used to increase the amount of dietary fibre or as, in particular, prebiotic ingredient in bakery products. In food inulin presents a hydroscopic property and is able to reduce the available water contents during gelatinization of starch, causing carbohydrates to be less absorbed, leading to a lower glycaemia rate.

Rheological measurements are often used to predict the behaviour of wheat dough during processing and the quality of the final product (bread). Mixes of wheat flour (T650) and inulin from chicory (Deracel CF 20, Dera Food Technology, Belgium) were prepared by substitution of wheat flour by 5 %, 10 %, 15 %, 20 % and 25 % of inulin. The rheological properties of the dough produced from these flour mixes were studied by Farinograph – E, Brabender OhG, Duisburg, Germany (ICC-Standard 115/1, 1992, AACC Method 54T21, 1995). The effects of the inulin addition on water absorption, dough development time, stability and degree of softening were established. Low addition of inulin (5 % and 10 %) decreased water absorption capacity of mixes in comparison to wheat flour; however, higher addition of inulin (15 % - 25 %) proportionally increased the water absorption capacity of mixes. The addition of inulin caused changes of the rheological parameters of the recorder curve. 10 % and more addition significantly extended development time and there were two peaks of consistency on the farinogram which is a non-standard shape. With increased addition of inulin the resistance to deformation has grown and the dough was difficult to process. Mixes with higher than 15 % of inulin addition were unsuitable for making bread.

Within bread-making procedure 1000 g wheat flour (T650) or flours mixes with inulin were used for the preparation of test breads. Bread quality was evaluated 24 hours after baking: weight, volume, specific loaf volume and volume efficiency, bake loss, height/width ratio and crumb acidity. Bread volume, the most important parameter decreased significantly with inulin addition. The sensory evaluation was conducted according to a sensory descriptor. Our results suggest that a level of 5 % inulin addition leads to a production of a functional bread of high sensory acceptance and a level of 10 % inulin produces a bread of a satisfactory sensory acceptance. Bread with a level over 10 % of inulin addition was evaluated as unsatisfactory.

**Keywords:** *farinograph evaluation, inulin addition, bread test*

## **Preliminary observations on the effects of milk fortification with conjugated linoleic acid in yogurt preparation**

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Conjugated linoleic acid (CLA) is a group of positional and geometric isomers of conjugated dienoic derivatives of linoleic acid. The cis-9,trans-11 isomer of CLA, also known as rumenic acid, accounts for 90% of the total CLA intake and is mostly found in beef and dairy products. The cis-9,trans-11 isomer in CLA has been considered to be the most biologically important in terms of anticarcinogenic activity, protection against artherosclerosis, cachexia, and treatment of noninsulin-dependent diabetes.

The level of CLA in milkfat commonly falls between 3 and 6 mg/g. To receive the recommended 3 g/day, approximately 30 servings per day (cca. 250 mL) of milk with naturally increased levels of CLA would be necessary. Direct fortification of milk with CLA oil is a more feasible and realistic solution to obtain maximum health benefits. The aim of this paper is to determinate in which way can influence the CLA fortification the rheological properties of the obtained yoghurt product, because this information may correlate with the yoghurt sensory attributes (thickness, stickiness). For this issue we prepared on laboratory scale two different samples of yoghurt with and without CLA and the obtained data was compared with a brought yoghurt from commerce. The rheological study was effectuated on an Anton Paar Physica MCR 501 rheometer. Sample with CLA presented the most stable mechanical structure, an extended linear viscoelastic (LVE) domain and a smooth feeling as compared both with sample without CLA and commercial yogurt.

**Effect of high pressure processing on rheological properties and pectinmethylesterase activity of Aloe vera (*Aloe barbadensis miller*) juice**

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Effect of pressure level (60-750 MPa), dwell time (3-30 min) and pH (2.32-5.68) on rheological properties and pectinmethylesterase (PME) enzyme activity of aloe vera juice was studied using response surface methodology (RSM) based on central composite rotatable design (CCRD). Rheological analysis of aloe vera juice showed shear thinning behavior and was well described by Power law. Magnitude of consistency index, flow behavior index and yield stress ranged in 59.75-78.9, 0.64-0.7 and 0.12-0.19 (Pa) respectively. PME activity ranged between 0.036-0.06 (PEunit/ml). A quadratic model was developed for rheological parameters and PME activity; it was found that pressure level had a significant effect on all the response variables ( $p < 0.05$ ) followed by pH and dwell time.

**Keywords:** *Aloe vera, rheological properties, high pressure processing, power law*

**Session 4.**

**Avalanche Dynamics in Rheology  
and Phase Transitions**

## **Twinning in Strained Ferroelastics: Microstructure and Statistics**

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The dynamic evolution in ferroelastic crystals under external shear is explored by computer simulation of a two-dimensional model. The characteristic geometrical patterns obtained during shear deformation include dynamic tweed in the elastic regime as well as interpenetrating needle domains in the plastic regime. As a result, the statistics of 'jerk energy' differ in the elastic and plastic regimes. In the elastic regime the distributions of jerk energy are sensitive to temperature and initial configurations. However, in the plastic regime the jerk distributions are rather robust and do not depend much on the details of the configurations, although the geometrical pattern formed after yield is strongly influenced by the elastic constants of the materials and the configurations we used. Specifically, for all geometrical configurations we studied, the energy distribution of jerks shows a power law noise pattern  $P(E) \sim E^{-(\gamma-1)}$  ( $\gamma-1 = 1.3-2$ ) at low temperatures and a Vogel-Fulcher distribution  $P(E) \sim \exp(-E/E_0)$  at high temperatures. More complex behavior occurs at the crossover between these two regimes where our simulated jerk distributions are very well described by a generalized Poisson distributions  $P(E) \sim E^{-(\gamma-1)} \exp(-(E/E_0)^n)$  with  $n=0.4-0.5$  and  $\gamma-1 \approx 0$  (Kohlrausch law). The geometrical mechanisms for the evolution of the ferroelastic microstructure under strain deformation remain similar in all thermal regimes whereas their thermodynamic behavior differs dramatically: on heating, from power law statistics via the Kohlrausch law to a Vogel-Fulcher law. There is hence no simple way to predict the local evolution of the twin microstructure from just the observed statistical behavior of a ferroelastic crystal. It is shown that the Poisson distribution is a convenient way to describe the crossover behavior contained in all the experimental data. In addition, We find that shear on materials with soft bulk moduli have much higher junction densities than those with hard bulk moduli. Soft materials also show an increase in the junction density with diminishing sample size. The change of the complexity and the number density of twin boundaries represents an important step forward in the development of 'domain boundary engineering', where the functionality of the materials is directly linked to the domain pattern.

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**Keywords:** twin boundary density; junctions; driven dynamical system; soft and hard materials.

## **Onset of Irreversibility and Chaos in Amorphous Solids Under Periodic Shear**

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An important aspect of the physics of amorphous solids is the onset of irreversible behavior usually associated with yield. Here we study amorphous solids under periodic shear using quasi-static molecular dynamics simulations and observe a transition from reversible to irreversible deformation at a critical strain amplitude. We find that for small strain amplitudes the system exhibits a noisy but repetitive limit-cycle, similar to return point memory. However, for large strain amplitudes the behavior becomes chaotic (shows sensitivity to initial conditions) and thus irreversible. We show that the chaotic behavior is a result of the shear band instabilities/avalanches that arise for large strains and the convective displacement fields they create.



## **On the Mechanical Loss in Ferroelastic Shape Memory Alloys**

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One of the most important high damping materials is shape memory alloy, which undergoes a martensitic transformation. The high mobility of twin boundaries gives rise to high mechanical loss. Previous study have shown that the martensitic transformation is associated with one high internal friction peak. After the phase transition, the internal friction still shows a non-zero value.

In the present study, we have investigated the mechanical loss of shape memory alloys at different frequencies by Dynamic Mechanical Analysis (DMA) measurement. Our results show that the pure alloy with no defect doping shows a frequency-independent damping plateau over a wide temperature range after the martensitic transformation. When the alloy is doped with defect (a small amount of hydrogen atoms in interstitial site), there is an extra high damping peak besides the damping plateau. Previous study have suggested that such damping peak can be ascribed to the interaction between twin boundaries and hydrogen atoms under external stress during DMA measurement.<sup>[1]</sup> More importantly, the value of damping plateau is almost same with the one with no hydrogen doping and it does not depends on the frequency either. This indicates that the defect (hydrogen) doping does not change the damping plateau in martensitic state, which results from the intrinsic hysteretic movement of twin boundaries. Our experiment results are consistent with previous simulation results that even the pure ferroelastic system with only one needle twin still results in energy dissipation.<sup>[2]</sup>

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**Keywords:** *Mechanical loss; twin boundary; martensitic; damping plateau;*

## **Friction in ferroelastic and martensitic materials**

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Each elastic hysteresis has two branches: first the elastic branch which represents the elasticity of the bulk material and is hard. The second branch topologically connects the elastic branches and relates to the change of micro-structures under applied elastic strain fields and is soft. The dynamics of the soft branch is viscous and follows the laws of fluid dynamics rather than classic elasticity theory. When soft branches are followed dynamically under increasing external strain (~hard boundary conditions) over very long time scales one observes spontaneous changes of microstructures such as twins and anti-phase boundaries. Each change leads to a sharp energy absorption over a very small time interval. The distribution of these energy changes (~jerks) are related to avalanches of strain relaxations. The probability distribution of the jerks was found experimentally to follow a power law with energy exponents between 1.3 and 2. Experimental results and observations from Molecular Dynamics simulations of large systems will be discussed.

## **Avalanches in shock induced plastic deformation and phase transition: A molecular dynamics study**

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Materials dynamics, especially the effects of plasticity and phase transformations under extreme dynamic condition, is a topic of broad scientific and technological interest. However, the statistical behavior of shock induced plastic deformation and phase transition remain unclear so far. It is also not clear how do they differ from that under quasi-static loading. In this talk, by means of equilibrium and nonequilibrium molecular dynamics simulations, we compared the avalanches (or jerks behavior) of uniaxial compression induced plasticity (in Cu single crystal) and martensitic transformation (in Fe single crystal) in shock and quasi-static condition. Our results show that shock can dramatically change the statistical behavior of plastic deformation and phase transition. Under quasi-static uniaxial compression, the distributions of energy jerks of both dislocation in Cu and martensitic transformation in Fe follow the exponential behavior, indicating a thermally activated behavior under quasi-static condition. In contrast, the distribution of the energy jerks of Cu and Fe single crystal follows a power law relationship under shockwave compression, this means that shock can change the statistical behavior of plastic deformation and phase transition from thermally activated to athermal behavior. We suggest that the underlying reason could be related to the higher nucleation rate of martensite or dislocation slip in shock condition.

**Keywords:** *statistical behavior; quasi-static compression; shock; martensitic transformation; dislocation slip.*

## **Influence of strongly anisotropic materials crystal structure anisotropy on a structure and property of their basis ceramics, synthesized in the solar furnace.**

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For ceramic materials synthesis the solid state reactions method is mainly used. "Fusion" technologies application is more limited. Ceramic materials synthesis "fusion" technologies have a advantage before "solid state". Completeness of a reactions a between initial components allows to realise completeness of synthesis and homogeneity of a target materials. At subsequent heat treatment of their morphology operate. Restriction of the technologies based on synthesis by "fusion" methods using is of their high power consumption appreciably is connected. Power resources restriction and ecology preservation vital necessity allow a search and a development of a new technologies. This technologies on renewed, ecologically pure energy sources is based. From this point view solar energy for functional materials production technologies use is interest. Solar furnace synthesis conditions provides to high cleanliness, homogeneity, oxygen stoichiometry. Melt cooling conditions of quenched material to morphology big influence have been rendered. Melt cooling speed change allows to amorphous, glasse-crystal and crystal states receive. At the melt quenching process, because of the big temperature gradient, high texture in target materials and in target ceramics remains. The texture with phases mutual orientation character is connected. At annealing process between grains borders a pressure concentration arises. Pressure can cause disintegration of a target material. Research of strongly anisotropic materials of pseudobrookite-type structure ( $\text{Al}_2\text{TiO}_5$ ,  $\text{MgTi}_2\text{O}_5$ ,  $\text{Fe}_2\text{TiO}_5$ ) and superconducting compositions-homological  $\text{Bi}_{1,7}\text{Pb}_{0,3}\text{Sr}_2\text{Ca}_{(n-1)}\text{Cu}_n\text{O}_y$  ( $n=2-20$ ) have been synthesised in the solar furnace, along of a crystal structure greatest expansion direction high texture formation has shown. Grain size critical dimension in all compositions 120-150 microns has made. These parametres excess led to disintegration of compositions. Texture undesirable influence on a pseudobrookite-type ceramics thermomechanical properties by no strictly focused microstructure has been eliminated. For superconducting ceramics of critical parametres increase to kept of the texture by annealing at gradient conditions.

**Session 5.**  
**Experimental Study and Modeling  
of Complex Flows**

## **Operability Coating Windows in Slot Die Coating Processes**

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Flow dynamics in coating bead region and operability coating windows in slot coating process, which has been importantly implemented in IT industries manufacturing secondary batteries, flat panel displays, etc., have been investigated via modeling, simulation, and visualization experiments. Viscocapillary models for predicting the operability window have been developed, exhibiting good agreement with 2-D and 3-D models and experiments. Also, various issues on slot coating flows will be introduced in this talk, including the effect of sloping die configuration on the operability window using 2-D model, coating flow behavior from internal and external die regimes via 3-D simulation, wetting characteristics with respect to contact angles, and dynamics of multi-lane stripe coating.

**Keywords:** *slot coating, dynamics, operability windows, coating bead flow*

## **Dynamic Analysis of Marine Drilling Riser for Deepwater Environment**

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In recent years, exploration and production activities of oil and gas in offshore have increased in deep and ultra deep water. As one of the key elements in offshore infrastructure, deepwater riser technology plays an important role in offshore development. The drilling riser is the key element for drilling in deepwater. A drilling riser is a tube that can be made of steel, aluminum, titanium or composite materials that is used to conduct drilling, running and setting casing, cementing and coring operations through the seawater column.

The development of deepwater oil and gas reserves constantly faces the challenge to reduce costs of all components and activities. A drilling riser is a conduit that provides a temporary extension of a subsea oil well to a surface drilling facility. With an increase in drilling operations in harsh environments, drilling riser requirements and limits have become more onerous due to uncertainties involved in response prediction and prolonged drilling programs. A high level of understanding is required of the response of the system to various conditions, and the design issues that govern the system.

The rheological properties of drilling mud such as density, viscosity, power law index, etc. influence on the result of riser analysis. The mud density dependences on the drilling riser analysis results and two mud density values considered are 8.56 ppg and 15 ppg. Also, Static and dynamic analysis, especially VIV (vortex induced vibration) analysis of drilling riser system have been carried out for the long-term fatigue performance evaluation and development of drilling riser joint. This paper presents an overview of the engineering challenges faced in designing drilling riser system for drill ship in water depths of 3,000m.

**Keywords:** *Offshore drilling, Marine drilling riser, Riser analysis, Drilling mud*

## **An Experimental Study on the Cuttings Transport in Inclined Slim-Hole Annulus**

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Among the various industrial unit operations involved with multi-phase systems, agitation of solid-liquid systems is quite commonly encountered such as catalytic reactions, drilling operation of oil well, etc. Although there are many industrial applications of solid-liquid mixture flows in technology, the available knowledge about particle flows is not complete due to the difficulties encountered in analyzing these complex systems.

In directional drilling, it is difficult to adjust and control the cuttings, so it is very important to evaluate the flow characteristics of a drilling flow field. In this study, solid-liquid two-phase flow experiments have been carried out in Newtonian and non-Newtonian fluids for hole inclinations from vertical to 75 degrees, flow velocities from 0.33 m/s to 0.66 m/s, particle concentration from 4 to 16 %, and pipe rotations from 0 to 400 rpm. Pressure drop within the test section, and particle volume fraction are measured for the above test conditions. These quantities were influenced by particle concentration within the flow, pipe rotation, flow volume, and inclination of the annulus. Moreover, empirical correlations were developed for estimating friction coefficient and particle volume fraction inside annulus. The new correlations generated in this study are believed to be very practical and handy when they are used in the field.

That is, user friendly empirical correlations for estimating stationary bed thickness and friction coefficient were developed using experimental data. Results show that correlations may estimate with an error of  $\pm 20$  in most cases. Pipe rotation drastically decreases frictional pressure loss inside inclined annulus if there are cuttings present. In terms of Froude number and feed concentration of non-Newtonian fluids, 0.2% and 0.4% CMC solutions show a similar pattern with water. But there is a little difference.

**Keywords:** *Solid-liquid mixture flow, Slim-hole annulus, Particle concentration, Pressure loss*



## **Viscoelastic Behavior of Bitumen in Dynamic Control Umbilicals**

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By combination of elements like hydraulic tubes, electrical and optical signal cables and electrical power cables, umbilicals provide remote control of subsea oil and gas wells. For extra tensile strength and outer protection the umbilicals may be armoured by galvanized steel wires. Dynamic umbilicals are terminated at offshore platforms or vessels. They are exposed to tension and bending variations caused by waves and vessel motions. As such a project specific analysis is normally required to verify that a given dynamic umbilical design with all its elements is able to withstand a service life of typically 20-30 years.

Understanding how forces are transmitted between the elements in an umbilical is vital for correct calculation of a dynamic umbilical design life. Cross section analysis is therefore performed by a finite element method program specially designed for this purpose. This software takes into account all effects in an umbilical cross section, including friction between elements. For solid elements, commonly known friction factors may be applied. However, for the steel wire armour which is corrosion protected by bitumen, the friction factors are not valid. The force transmission between the steel wires is not governed by friction but by the viscoelastic properties of bitumen. Hence, the friction force is not only a function of contact pressure but also of sliding velocity.

A project was therefore initiated with the aim of describing the force transmission between bitumen and steel wires for typical umbilical conditions. Material testing has included characterization by a controlled stress rheometer, tensile and cyclic tensile testing at various temperatures, speeds and geometries. On basis of these tests, a calculation model describing the mechanical behavior of bitumen in cyclic movement is suggested. This model may later be implemented in the umbilical cross section analysis program.

**Keywords:** *Rheology, modelling, bitumen, dynamic, umbilicals*

## **Thixotropic and Shear Banding Flow Effects in a Continuous Squeeze Flow of a Structured Fluid**

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In this work, the viscoelastic behavior of a complex structural liquid in a continuous squeeze flow is analyzed. This flow is generated by allowing a continuous flow of liquid into the narrow gap between two circular plates through the lower plate. In order to characterize our complex liquid, the Boek model is used (BM). The BM model separates the stress contributions of the structured fluid and the solvent. The solvent is modeled as a purely Newtonian liquid, and the complex fluid is characterized with the upper convective Maxwell equation coupled with a kinetic equation that describes the changes of the structures due to the relaxation and kinetic mechanisms respectively. In order to solve the set of non-linear partial differential equations, a non-dimensional perturbation scheme is suggested in terms of a small parameter, which is the ratio between two characteristic length scales (radius of the disc and gap separation between the disc). To zero order in the perturbation parameter (neglecting the inertial mechanisms of the momentum equation), it is found that the normal force on the upper disc is directly related to the structure of the fluid, which is a function of the Weissenberg number. The shear banding, thixotropy, shear-thinning, shear thinning, yield stress and concentration effects are analyzed through a group of characteristic dimensionless numbers associated to structural, kinetics and viscoelastic mechanisms respectively. To first order, the effects of the rupture and structural mechanism play an important role in the elasticity. The present theory, model and computations contribute to the evolving fundamental understanding of lubrication systems through rheology and flow systems

## **Metallurgical processes to extract gold from ores and concentrates in the aquatic environment**

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To determine the parameters of the process of opening for mechanical activation of a planetary centrifugal mill were processed arsenopyrite ore and ferrite in the aquatic environment. We use two types of raw materials: arsenopyrite-1 and arsenopyrite-2.

The main parameters of the process of determining the effectiveness of the treatment is the treatment, the degree of loading and the ratio of crushed minerals from the liquid. To determine the optimum time of activation treatment conducted at various time minerals modes from 5 to 30 minutes in increments of 5 minutes and a different ratio of grinding bodies and processed mineral. The ratio of liquid: solid is 2:1. The ratio of grinding media and the material (Balls: Solid) is 2:1, 4:1, 6:1 and 10:1. The resulting slurry was clarified, the solution was decanted and the pH were determined and the amount of sulfate ion in solution after activation sulphides. Found that the filtrate after activation of hydrogen sulphide has a distinct character. Depending on the processing parameters, the pH of the solution was varied from 7.0 to 6.0, which implies that there was a partial oxidation of sulphide minerals contained by the reaction:  $2\text{FeS}_2 + 6\text{H}_2\text{O} + 6,5 \text{O}_2 = \text{Fe}_2\text{O}_3 + 2\text{H}_2\text{SO}_4 + 2\text{H}_2 \uparrow$ . The appearance of hydrogen in air-tight drums have a certificate of participation in the water as the oxidant. The main indicator of the oxidation process is the residual sulfur in the sediments. XRD data indicate a structural change in the crystal lattice and the formation of new phases in arsenopyrite  $\text{FeS}_{1-x}$  and arsenite in small amounts when activated 20 and 30 minutes, the ratio of Balls: Solid is 4:1. The more intense was the mechanical effect on the minerals, the great changes in their crystal lattices. In ferritic grades after 30 minutes of activation, a new phase is  $\text{Fe}_2\text{O}_3$ . Maximum transition sulfur solution for arsenopyrite-1 occurred after treatment for 30 minutes and the ratio of Balls: Solid = 4: 1 and was 69.32% for the arsenopyrite-2 under the same conditions is 71, 77%. The increase in the ratio of grinding media did not lead to a deeper degradation of sulfides. The filtrate settling pulp after activation were analyzed for arsenic content. Number of arsenic in the filtrate depending on the treatment time is from 0.24 to 0.54 mg / l. Low transition arsenic filtrate shows the binding of arsenic compound in the form of sparingly ferric arsenate. This confirmation of this is the increase in the relative content of arsenic in the sediments.

**Keywords:** *recovery, gold-bearing concentrate, gold ore, mechanical activation, water environment*

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## **Session 7.**

# **Multiscale Mechanics of Materials**

## **Multiscale modelling heat transfer in two-phase composites**

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Two-phase Ceramic Matrix Composites (CMCs) or Metal Ceramic Composites (MCCs) have many applications as different parts of advanced technology structures like rockets, aircrafts for military or civil applications, nuclear power and wind power plants. A very good example of CMCs is a ceramic material created from a mixture of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$ . They are applied as thermal barriers against sudden temperature changes. Examples are Thermal Barrier Coatings (TBC) of turbine blades and vanes of engines as well as protections of the reactor elements in nuclear power plants. A typical example of CMMs is cermet WC/Co, i.e. the metal–ceramic composite composed of brittle grains WC joined by the plastic binder Co.

The above mentioned composites are subjected to thermo-mechanical loading under real working conditions as the structural parts of engineering constructions. It is necessary therefore to perform an analysis of heat transfer throughout the internal structure of a composite, which includes: grain shapes, interfaces and porosity. The description of the heat transfer is done at mesoscale level by introduction of the Representative Surface Element (RES) including a representative number of grains and interfaces of the composite. Heat passes through the RSE, creating stress concentrations due to misfit of the thermal expansion coefficient of the both phases. Numerical analysis with the application of the commercial code ABAQUS allows for localization of efforted places within internal structure of the composite. They are sources of damage at the interfaces of different phases. With the assumption, that the composite is homogeneous one can estimate macroscopic scale parameters of the heat flow through simple and complex internal structures of composites.

**Keywords:** *multiphase modelling, two-phase composites, heat transfer, stress concentrations*

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- 2) This work was financially supported by Ministry of Science and Higher Education within the statutory research number S/20/2013.

## **Structure, phase content and mechanical properties of aluminum with hard particles after shock-wave compaction**

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Structure and properties of materials based on aluminum, reinforced by hard particles carbon and alumina after shock-wave compaction was studied. It was found that explosion compaction of aluminum powder and powder mixtures Al +10 wt.% C (in the form of detonation diamonds) and Al +10 wt.% Al<sub>2</sub>O<sub>3</sub> produces samples with a density close to the theoretical. X-ray diffraction studies showed that the samples with the addition of carbon and aluminum oxide formed two-phase aluminum state with different structure parameters. In X-ray profiles, consisting of the strong-intensity line of aluminum and at smaller angles there was a weak-intensity reflection. The CDD of crystallites of the strong-intensity line in all cases were about  $80 \pm 10$  nm and this value is close to the CDD of aluminum powder in the initial state. CDD of weak-intensity reflection for the samples with detonation diamonds was about  $13 \pm 5$  nm and with aluminum oxide nanopowder -  $8 \pm 5$  nm. In this case, the lattice parameter of this phase was increased by 0.5%, which may be an indication that in the surface layers of the samples there are formed residual compressive stresses, the evaluation of which gives the value of 350 MPa. It was shown that the hardness and yield strength of materials after shock-wave treatment increased almost 10-times higher as compared to commercially pure aluminum.

## **Plasticity in polymer glasses: from mesoscopic dynamics to macroscopic response**

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Based on the concept of localized shear transformation zones (STZ), as initially proposed by Spaepen [1] and more recently refined by e.g. Falk and Langer [2,3], a thermodynamically consistent model, using GENERIC [4,5], for the viscoplastic deformation of glassy polymers is developed. The approach consists of a dynamic description of macroscopic viscoplasticity that is enriched by the evolution of number density and internal structure of the STZ, i.e. nucleation/destruction and average orientation, for detailing the origin of viscoplastic flow. To close the constitutive relation an assumption for the relaxation of zone reorientation is needed.

Elaborate ansatzes for the viscosity function have been made in literature that allow for accurate modeling of complex deformations [6,7]. In this work we do not attempt to improve on these successful phenomenological ansatzes, but use the stress activated transition on the STZ level to explain the observed macroscopic response. The microscopic model expresses typical features, i.e. the dependence of yield stress on deformation rate and age.

The activation of STZ upon deformation and their subsequent internal re-arrangements are treated as two distinct processes. The detail of the model permits to relate it to small-scale information obtained from experiments and/or atomistic computer simulations. Typical features we include in the model are the STZ-activation energies and an approximate STZ volume. We will demonstrate the applicability of the model on our model material that is polycarbonate.

**Keywords:** *structure-property relation, polymer glasses, two-scale model, viscoplasticity*

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## **Numerical Simulation of Dynamic Loading to Thin-Walled Corrugation Tube Aluminum 6061-T6 Alloy**

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The requirement of design of tubular structure is major attention for structural components such as automobile, aircraft and marine transportations. The present paper deals with the implementation of the finite element to simulate the crash behavior and energy absorption characteristics of aluminum alloy extrusion thin-walled tubes of radial corrugation cross-section subjected to high velocity of axial impact loading. The collapse procedure is successfully simulated and the verification of numerical model was compared with other analysis with previous investigation. Both of thin-walled metallic structure aluminum alloy extrusion was describe numerically and that introducing radial corrugation along a shell element generator together with a proper advanced metallic material will enhance the crashworthiness performance of absorbed energy device units. On the other hand, the comparison was carried out within both of the result in term of dynamic load and dynamic absorbed energy versus displacement. Results indicated that the thin-walled energy absorption capability was affected significantly by varying the radial corrugation and aspect ratios. It is also found that as the number of radial corrugations increase, the amount of absorbed energy significantly increases.

**Keywords:** *Energy Absorption, Thin-Walled Corrugation Structure, Finite Element Analysis, MALAYSIA*



## **Fluctuation-based flow-rule for viscoplastic flow and rate-dependent strain hardening of solid polymers**

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The yielding behavior of anisotropic polymer solids is studied from a modeling perspective. To account for the dynamics of yielding, a fully 3D elasto-viscoplastic setting is adopted [1], in which the key issue is the formulation of the flow rule, i.e. the relation between the plastic strain rate tensor, the applied load, and the inherent anisotropy. Using a generalized fluctuation-dissipation theorem, we obtain the flow rule for transversely isotropic materials from symmetry considerations on fluctuations of the elastic deformation gradient [2,3]. It is pointed out that this procedure does not rely on classical yield criteria (e.g. [4]) or on the associated flow rule [5,6].

The usefulness of the fluctuation-based flow rule in the elasto-viscoplastic model is demonstrated by applying it to experimental yield data of uniaxially oriented isotactic polypropylene [7]. The dependence of the yield stress both on deformation rate and on drawing direction is captured at the same time. It is also exemplified on these experimental data that the the common associated flow-rule is inherently inconsistent, which is not the case for the fluctuation-based flow rule. Finally, it is shown how the fluctuation-based flow rule can be employed to describe rate-dependent hardening in polymer solids [8], namely through continuously increasing the anisotropy during plastic deformation.

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**Keywords:** *generalized fluctuation-dissipation theorem, anisotropic yielding, plastic flow-rule, viscoplasticity, rate-dependent strain hardening*

## **FEM Simulation of the Size- and Constraining Effect in Lead- Free Solder Joints in the Framework of Strain Gradient Elasticity**

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It is a well known fact that the mechanical properties of miniaturized solder joints cannot simply be deduced from the behavior of bulk solder material. Instead, a pronounced size effect is observed leading to the consequence that smaller samples show higher strength. Aside from effects of microstructure, the material behavior is governed by geometry effects and intrinsic size effects. Geometry effects are a result of constraints occurring along interfaces to the stronger base material. Intrinsic size effects occur when sample dimensions approach an internal length scale of the material. In the present study, these phenomena will be studied in the framework of strain gradient elasticity.

Strain gradient elasticity may be considered as a deviation from Hooke's law which occurs when sample dimensions approach a small scale. While Hooke's law relates stresses to strains, strain gradient elasticity additionally considers higher order stresses which are related to strain gradients. The proportionality of higher order stresses and strain gradients is described by a bending modulus which has the dimension of a force. Since the dimension of the bending modulus is different from that of ordinary elastic constants, a size effect occurs when a sample is deformed inhomogeneously. Thereby, small samples show an increased stiffness against inhomogeneous straining. The reason for this behavior is provided by the fact that a strain gradient leads to a bending of the microstructure. But since the microstructure wants to preserve its integrity, stiffness against bending is the consequence.

We here propose a version of strain gradient elasticity which is slightly different from the theory published by Toupin and Mindlin in the 1960s. Although we derive the expression for the internal elastic energy in similar fashion, we consequently require that the stress tensor of our theory is symmetric in static equilibrium. This theory is implemented in the commercial FEM code ABAQUS through user subroutine UEL. Strains and strain gradients are calculated from the interpolation functions of the elements. Residual forces counteract the deformation introduced by strains and strain gradients. The magnitude of nodal forces is obtained from the condition that the work done by the external forces must coincide with the energy stored in the element. The boundary conditions for the strains at the borders of neighboring elements are satisfied with use of a novel overlapping mesh technique.

When the theory is applied to solder joints, one sees that it has a major influence on stress concentrations arising at material transitions at the surface. While classical continuum mechanics predicts a singularity of stress there, our approach of strain gradient elasticity reduces the stress to finite values. Moreover, the FEM simulations predict a size effect.

Finally, the theoretical model is applied to experiments performed with lead-free Sn-Ag-Cu solder joints. A three-dimensional digital image correlation system (DIC) with high spatial resolution was used for *in situ* measurement of the strain field during tensile testing. DIC is a non-contacting optical technique which compares images of the deformed sample with an initial reference image. With use of two cameras, in-plane and out-of-plane displacement fields of the object's surface are determined. Thereby, even extremely inhomogeneous deformation fields may be captured. Thus, the experimental findings are compared to the theoretical predictions of strain gradient theory. In fact, the deformations near the interface of solder and base material are in better accordance with strain gradient theory than with predictions of classical continuum mechanics.

**Keywords:** size effect, internal length scale, strain gradient elasticity, solder joints, singularity removal, digital image correlation

## **Theoretical and experimental studies of bending deformation in fivefold twinned silver nanowires**

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Metallic nanowires are of great interest due to their potential applications in different fields including nanophotonic devices [H. Wei *et al.*, Nanophotonics 1 (2012)], (flexible-) nanoelectronics [H. C. Ko *et al.*, Nature 454 (2008)], biological nanosensors [H.-Y. Lin *et al.*, Analytical Chemistry 80 (2008)], nanoelectromechanical systems (NEMSs) [E. F. Arkan *et al.*, Journal of Micromechanics and Microengineering 21 (2011)], etc. Deformation of nanowires under tensile and compressive loading has been studied extensively using atomistic tools while little is known about deformation mechanisms under bending. Bending of nanowires into different shapes is desirable in order to incorporate them into micro- and nanoscale devices. Elaboration of bending mechanisms is essential for improving the properties and finding new applications for metallic nanowires.

We have previously reported experimental setup for measuring Young's modulus and bending strength of nanowires *in situ* using nanomanipulation techniques inside a scanning electron microscope (SEM) [B. Polyakov *et al.*, The European Physical Journal B 85 (2012)].

The purpose of current work is to elaborate the mechanical response of fivefold twinned silver nanowires with pentagonal cross section under bending deformation, using molecular dynamics simulation. Relationships between atomic arrangement, defect formation and mechanical properties are studied. Results of the simulation are compared to those obtained by experimental techniques similar to the ones in our previous report (B. Polyakov *et al.*).

**Keywords:** *molecular dynamics simulation, fivefold, twin boundary, nanowire, bending*

## **Modeling strain and dielectric hysteretic type dependences in polycrystalline ferroelectrics by methods of two-level continuum**

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Polycrystalline ferroelectric materials are widely used in engineering to create sensors and actuators for various purposes. They may be made in the form of bulk, flat, linear or thin-film elements. During the process of polarization, as well as the application of external electric fields or mechanical stresses above the threshold, in such materials take place the irreversible processes that change the structure of the material. In this case, the electrical and mechanical response of the material presents the hysteretic type dependencies. The finite element method are used for the numerical analysis of transducers, in which the construction of constitutive relations is one of the key points. In this paper we present a model describing the irreversible processes in polycrystalline ferroelectric materials hysteresis type [1]. It is a kind of two-level model of a continuum in which each particle seen as a continuum of the first level, and a separate ferroelectric domain is considered as a particle of the second level. Construction of the model is carried out in three stages. In the first stage, the switching process of domains is modeled under the influence of efficient electrical and mechanical fields of Weiss. Thereby here we neglected the mutual influence of domains on the process of switching. The dependences of the limiting polarization and strain are obtained in the particle of the first level continuum by averaging over all switched domains, i.e. particles of the second level. At the second stage we estimate the energy required for breaking mechanisms of pinning domain walls, which is averaged by an arbitrary macro continuum. In the third stage we estimate the work of the electric field and mechanical stress associated with losses in the irreversible process of deformation and polarization. Evaluation of this work is also done for any macro continuum. Finally, to take into account the influence of neighboring domains on the process of polarization and deformation we derive the equation of the balance of energy on the macro level. Obtained from it relations are written in the form of differentials and represent the constitutive relations of the irreversible process of polarization and deformation. For solving of this system equations a method of successive approximations is proposed, which can also be regarded as a system equations in a finite differences. In this form this equations are used in the finite-element analysis of polycrystalline ferroelectric materials which subjected to intensive electric fields and stresses. The work was supported by the Russian Foundation for Basic Research (grant 12-01-00829-a, grant 13-08-01094-a).

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## **Experimental investigation of shock wave processes in solid and liquid paraffin and docosane.**

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Structure of compression front and mechanism of spall under tension at shock-wave action are determined by shock-wave parameters and condition of medium. It is of interest to investigate features of material response character on external influence at transition through melting temperature. The most clearly these changes can be observed in the metals. For example, above melting temperature two wave structure caused by elastic-plastic properties of metal vanishes away, the kinetic of spall destruction changes principally. Organic material behavior features investigated to a much less extent. The aim of this work is experimental investigation of shock-wave processes in solid and liquid paraffin and docosane.

Homogenized paraffin and docosane were chosen as objects of investigation. Paraffin is a mixt of saturated hydrocarbon molecules from  $C_{18}$  to  $C_{35}$  while docosane is saturated hydrocarbon  $C_{22}H_{46}$ . Compression pulse amplitude was changed from 0.2 to 2 GPa. Registration of free surface speed was made with VISAR laser interferometer. It was shown that phase transition of paraffin from solid to liquid state does not lead to appearance of any features on wave profiles. It was registered that spall strength of paraffin in solid state is equals of about 20 MPa and in liquid state it is equals of about 15 MPa. Insignificant difference can be observed either in the formation of spall pulse: its edge is much steeper in liquid form than in solid. Significantly different results were achieved in experiments with docosane. Registered spall strengths in solid and liquid state are the same and equals to about 20 MPa. In liquid phase speed profiles are similar to paraffin profiles, but in solid state the fact of dramatic change of compression pulse edge structure can be observed. In solid state with pressure less than 1 GPa can be seen two wave form with forerunner. Moreover the edge of the pulse “blurs” because of anomal compressibility. It means at first that solid docosane has an elastic property which leads to formation of forerunner. At second it means that at low pressures its compressibility is anomalous which is excluded opportunity of shock-wave formation.

**Keywords:** *shock-wave, spall, VISAR laser interferometer,*

## **Session 8.**

# **Rheological Fluid Mechanics**

## **INVERSE DESIGN AND OPTIMIZATION OF MAGNETORHEOLOGICAL DAMPERS AND RESONANCE MODELLING AND CONTROL**

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The potential of the variable yield stress of the fluid in magnetorheological damping devices modeled as a Bingham plastic to mitigate the destructive resonance effects imposed on various types of bodies by external forces is explored and a method to model and minimize resonant structural oscillations using magnetorheological dampers is presented. The response of the magnetorheological fluid flowing in a circular tube under a pressure gradient to the applied variable magnetic field is tailored to determine the optimum stress field in the fluid to mitigate resonance effects. The concept and the method developed can be applied to determine desired yield stress properties of the damper fluid in designing new magnetorheological devices to mitigate and possibly nullify resonance effects.

The related problem of the determination of the required constitutive properties of the magnetorheological (MR) fluid flowing in the damper is solved with the corresponding flow pattern via an inverse design process given a predetermined response of the damper to efficiently dampen a given load. The fluid is modeled as a Bingham plastic with time varying yield stress. Flow is governed by the continuously adjustable constitutive parameters of the MR fluid which are determined to generate variable resistance to flow to dampen the selected load efficiently. The method developed leads to the determination of the time dependent yield stress and consequently magnetic field variation necessary to achieve a specific displacement of the piston in the damper. The governing equations are solved for any time history of the dimensionless yield stress of the fluid which in turn is determined from the imposed response of the damper. The analytical tools developed are helpful in damper design leading to the optimization of the damping performance. The application of the method to resonance control and mitigation is illustrated.

## **Simulation of the variation in temperature in a material without and with default**

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In this article our principal study is the simulation of the variation in temperature in a solid material in absence and in presence the default, in particular a fracture on the level of surface of material, and to see how the default influences on heat transfer in a solid. The simulation is made by FLUENT software which permits us to solve the energy equation by finite volumes method.

**Keywords:** *heat transfer; simulation; material; default*



## **Numerical study of secondary flows of FENE rheological models in curved ducts**

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The study of non-Newtonian fluid flows in curved ducts has great interest due to the complex secondary flows generated in the cross-section of the ducts. This secondary flow is important to many industrial and engineering applications because it tends to increase mixing, mass and heat transfer, with either Newtonian or non-Newtonian fluids. In an attempt to better understand the development of those secondary flows and to assess the relative contributions of elastic and inertia forces many studies have been published assuming different rheological models to describe complex fluid behavior. Among the many available rheological models those which have received less attention in this type of flow are the FENE-type models (Finite Extensible Nonlinear Elastic), which are frequently employed in numerical simulation studies of viscoelastic flows because they give a reasonable description of the rheology of diluted and semi-diluted polymer solutions. An additional factor adding complexity to the generation of the secondary flows in curves is related to the geometry of the curved channel. Choosing either a square or a rectangular cross-sectional channel is known to be important for flow development because the flat walls, particularly those defining the outer concave wall of the curve, have been shown to enhance such secondary flow development.

The objective of the present work is to investigate, by means of numerical simulations, the development of secondary flow through a 180° curved duct having a square cross-section, of viscoelastic fluids described by three FENE-type models, namely the FENE-P (with the Peterlin approximation), the FENE-CR (with the Chilcott and Rallison approximation) and the FENE-MCR (with a modified form of the previous). The flow and constitutive equations were solved numerically with a finite-volume method, for various Reynolds and Weissenberg numbers and for a range of values of the dimensionless model parameters (namely, the  $\beta$ - retardation ratio and the  $L_2$ - extensibility parameter). The results of these simulations show complex changes of the three-dimensional flow, but also reveal similarities in the flow development for different rheological models.

**Keywords:** *rheology, curved duct, square cross-section, Finite Volume Method, viscoelastic fluids, FENE models.*

## Drag reduction of nata de coco suspensions in circular pipe flow

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There has been a growing interest in adding drag-reducing agents to reduce pipe friction and hence energy consumption. Additives such as polymers, surfactants, and fibers are well-known dragreducing agents. However, these additives have some limitations, such as being restricted for use in a closed-circuit pipeline system. Furthermore, they require careful disposal to prevent the drained solutions from contaminating the rivers and soil because their fibers are made of synthetic chemicals. In contrast, naturally occurring additives are considered to have a low environmental load. Therefore, research on drag reduction by naturally occurring additives is becoming increasingly important. Recently, nata de coco has been attracting attention as a drag-reducing additive. Nata de coco is expected to reduce drag by a large extent because its structure is similar to that of drag-reducing polymers; moreover, it also has the potential to exhibit low mechanical degradation because of the high mechanical strength of its filaments.

The dispersed state of fibers in a fiber suspension greatly influences drag reduction. However, the relationship between the dispersed state of nata de coco and drag reduction is currently unknown. Therefore, a major goal of research has been to reveal the influence of the dispersed state of nata de coco suspensions on drag reduction by measuring the pressure loss and visualizing the dispersed state.

Experiments were conducted by measuring the pressure drop of a fluid in straight circular pipes (inner diameter  $d = 15$  mm) in a pipeline flow loop. The tested nata de coco suspension, which was made from commercial sources, was immersed in tap water to remove sugar, mechanically pressed, and then dried. The test additives were prepared by mixing the dried nata de coco and tap water in a blender. Then, the suspensions were conditioned by injecting additives to the tap water circulated at a constant flow rate ( $Re = 22000$ ) through a pipe. The experiments were carried out by varying the scattering time to investigate the relationship between the time of scattering in the blender and the drag reduction. Fig. 1 shows the relationship between scattering time and drag reduction. Drag reduction is observed for scattering times of about 1 to 4 min, although it is not observed in the case of long (more than 5 min) or short (less than 1 min) scattering times. This implies that the dispersed state of nata de coco has a large influence on drag reduction. Subsequently, drag reduction increased by up to around 25% when the concentration of the suspensions (conditioned by injecting additives to the tap water circulated) was increased to 50 ppm. With regard to the mechanism of the relationship between the drag reduction and the dispersed state, Fig. 2 shows a micrograph of nata de coco fibers taken after an experiment in which drag reduction was observed. The figure reveals that a large network of fibers formed when drag reduction occurred. Such a network is not formed when drag reduction does not occur. Furthermore, drag reduction tends to increase as the scale of this network increases. Studies are presently being conducted to examine the conditions for network formation and the mechanisms of drag reduction through the use of high-speed cameras.

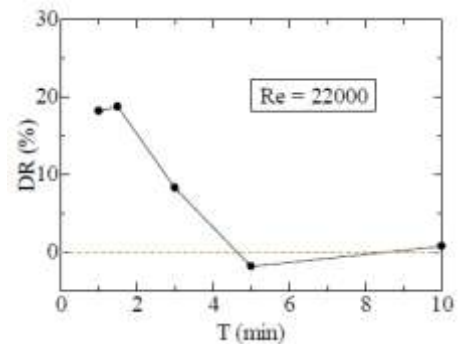


Fig. 1

Effect of scattering time on drag reduction

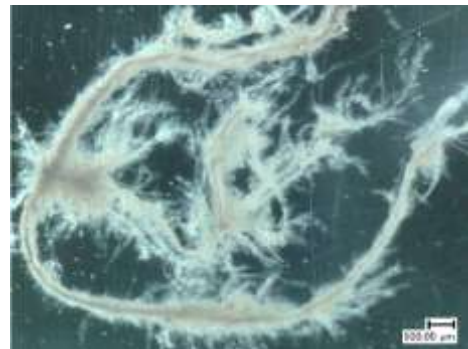


Fig. 2

Micrograph of nata de coco fibers

**Keywords:** Pressure loss, Visualization, Turbulent drag reduction, Nata de coco suspension

## A new numerical framework to simulate viscoelastic free-surface flows with the finite-volume method

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We present a numerical framework for computing the solution of viscoelastic flow problems with free surfaces, for the simulations of polymer processing technologies, e.g. extrusion, injection molding, etc. The basic governing equations to be solved are the Navier-Stokes equations for incompressible fluids, where the divergence of an additional viscoelastic stress tensor is used as a source term of momentum. The different components of the viscoelastic stress tensor are calculated from separate partial-differential constitutive equations. For highly elastic materials, the constitutive equations are prone to numerical instabilities (the well-known “high Weissenberg number problem”) which are remedied with the log-conformation change of variables.

Another numerical challenge is the resolution of the matrix equation system for the velocity and pressure unknowns, which is ill-conditioned. This difficulty is usually addressed in the literature by replacing the continuity equation with a Poisson pressure equation, derived by taking the divergence of the momentum equations (Chorin-Temam method). Furthermore, the resulting velocity-pressure system of equations is often decoupled with approximate projection methods, such as the fractional-step method, the generalized LU-block decomposition, or the semi-implicit methods (i.e. SIMPLER, SIMPLEC and PISO algorithms). Those methods involve sequential solution and correction steps of smaller systems of equations. However, they inherently produce decoupling errors, which eventually become dominant in the calculation of flows at low Reynolds number. Therefore those methods, although widely used, are not adapted to solve creeping flows such as in polymer manufacturing processes.

The novelty of our work lies in the use of a streamfunction scalar field as the primitive variable of the viscoelastic flow, in order to bypass the problem of the velocity-pressure coupling. The resulting matrix equation system for the streamfunction is similar to a bi-harmonic discrete operator, where the pressure unknowns have been eliminated.

All the governing equations are discretized with the finite-volume method, on a staggered structured orthogonal mesh. Additionally, the free surfaces of the material are tracked explicitly with the volume-of-fluid method. Test-case simulations are used to compare the results from the streamfunction-based and the pressure-based methods.

**Keywords:** *computational rheology, finite-volume method, viscoelastic flow, free surface flow, volume-of-fluid method, velocity-pressure decoupling, streamfunction method*

## **Colloidal Behavior of Aqueous Montmorillonite Suspensions in the Presence of Nonionic Polymer**

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In the petroleum industry, bentonite clay is a key in the formulation of water based drilling fluids (Cartalos and al. 1997, Coussot and al., 2004). Because of its extensive swelling in water, it forms a gel-like structure at relatively low concentrations (4-5% w/w) (Bekkour and al., 2005, Allal and al., 2009) due to the expansion of the clay platelets when saturated with Na<sup>+</sup> or Ca<sup>2+</sup> ions (Van Olphen, 1963). The clay is a texturing product and viscosifiant which is associated with polymers to assure the multiple functions: transport the cutting on surface, suspend the cutting when circulation of the drilling fluid is stopped, lubricate and cool the tool etc...

The influence of the interactions on the rheological properties was large studied in systems as the drilling fluids or model fluids (Rossi and al., 1997, Benna and al., 1999, Almedar and al., 2005, Manoratne and al., 2006, Ebagninin and al., 2006). Indeed, nanoparticles component of these suspensions, present a very important ratio surface on volume which should increase in theory, all their physical properties (viscosity, yield stress, elastic and loss modulus). In the case of clays, which are constituted by nanoplatelets in charge, we would have the possibility of having a gel of the mixture and thus a raised yield stress. Unfortunately, the dispersal of these platelets is problematic and the obtained properties are mediocre. So numerous research works, concern the improvement of the properties of these clays by addition of polymers (Ogata and al., 1997, Shen and al., 2002 and 2003), to adapt the composition of the fluid to drilling conditions and make a successfully operation.

In this article, we showed the influence of the polyethylene oxide (PEO) of molecular weight  $6 \times 10^3$  g/mol and  $2 \times 10^5$  g/mol on the rheological properties of the bentonite suspension (6%). The PEO presents an affinity for the bentonite particles gradually coated the clay particles, slowing down their kinetic aggregation. The reduction of particle-particle interaction took place until steady values of the yield stress. The stability of the suspension was enhanced with increasing polymer adsorption. The influence of this additive was investigated in terms of viscosity, yield stress, loss and elastic modulus which are principal characteristics of the drilling fluids. The analysis by X-rays diffraction also allowed understanding the rheological behaviour of the mixture bentonite-PEO. Indeed, the PEO can adopt diverse possible mechanisms: adsorption on the surface of particles, intercalation between the platelets of clay or favouring flocculation by forming bridges between particles.

**Keywords:** *rheology, suspension, viscoelasticity, bentonite, polyethylene oxide*

## **Rheological Model for Slipping Phenomena in Polymeric Fluids and her Application for Flows in a Channel with a Square Cross-section**

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Many fluid systems, including polymeric and bio-polymeric materials, reveal the anomaly of slipping often near the solid surface. The presence of such a wall effect leads to a violation of the hypothesis on attachment and the need to specify appropriate boundary conditions.

This anomalous behavior of materials in a plastic state (slurry, grease, fluids and polymer melts) at solid surfaces requires a comprehensive study of both rheological properties and calculating the flow parameters and characteristics of the processing equipment. In the first place, there are rather complex problems of determining the rheological characteristics of the material results according to the viscometric studies. The next stage is associated with specific problems on the motion of fluids which exhibit abnormalities in solid surfaces and the direct use the sliding velocities as boundary conditions.

This work is to specify explicitly the slip velocity at the wall, which is generally a function of stress at the wall, the geometric dimensions and temperature. And this dependence of slip velocity at the wall of these factors can be found from viscometric measurements.

Next considering the case of plane Poiseuille flow with allowance for slippage of the polymer material at the boundary, the system of equations of the modified model Vinogradov and Pokrovskii describes non parabolic velocity profile in the gap between parallel plates, which is confirmed by experimental data.

The dependences obtained can be used in the study of more complex flows. This is shown by the example of the calculation of the three-dimensional velocity profile of a nonlinear viscoelastic fluid in a channel with a square cross-section.

This work was supported by the Russian Foundation for Basic Research № 12-01-00033.

**Keywords:** *rheology, slipping phenomenon, polymer melts, mesoscopic approach*

## **Session 9.**

### **Rheology and Modeling of Hetero-modulus Hetero-viscous Complex Materials**

## **Investigation of aluminium-steel joints formed by explosion welding process**

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Explosion welding is a solid state welding process that is used for the metallurgical joining of metals. Explosion cladding can be used to join a wide variety of dissimilar or similar metals. This process uses the controlled detonation of explosives to accelerate one or both of the constituent metals into each other in such a manner as to cause the collision to fuse them together.

In this study, bonding ability of aluminum and steel with explosion welding was investigated. Experimental studies showed out that, aluminum and low carbon steel could be bonded with a good quality of bonding properties with explosion welding. In case of our process the clad metal was the low carbon contain unalloyed steel and the base metal was the aluminum sheet. It was found that, hardness of bonding interface and outer face of plates were increased because of deformation that was originating from impact the effect.

**Keywords:** *explosion welding, cladding, microstructure, microhardness*

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### 3D Diagrams for “Immiscibility Gap Technology”

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T-x-y diagrams with liquid immiscibility is employed widely for the solution of problem associated with a refractory compounds obtaining (borides, silicides and intermetallides). Besides the well-known flax method with only one solvent, the immiscibility gap method for the metallic systems was offered in the third millennium [V. Gurin, Yu. Grin', U. Burkhardt and M. Konovalov: Bull. Russ. Acad. Sci.: Phys. Vol. 73 (2009), p. 1386-1390]. In this method the synthesis and crystallization of compounds have place on the border of two solvents. As this technology includes many unknown details, computer models of phase diagrams for the ternary systems will be a useful tool for its understanding and improvement.

Computer templates is convenient to use for the simulation of phase diagrams with immiscibility surfaces, having a great quantity of topological modifications, produced by different arrangements and intersections of monovariant lines and immiscibility surfaces, and at the degeneration of solidus and solvus surfaces.

The use of computer model permits to find the error in the visualization of phase diagrams with liquid immiscibility gap. As an example, in [A. Prince: Alloy phase equilibria (Elsevier Publ. Comp, Amsterdam-London-N.Y. 1966) P. 218-223] the solvus surfaces in some systems have the orthogonal position of some lines on their perimeter with five points, but their projections have not the closed contour.

The computer model of T-x-y diagram with monovariant syntectic equilibrium with intermediate compound ( $\delta$ ) in one binary system permits to investigate the transition of syntectic equilibrium  $L_1+L_2 \rightleftharpoons \delta$  into the monotectic one ( $L_1 \rightleftharpoons L_2+\delta$  or  $L_2 \rightleftharpoons L_1+\delta$ ) in three-phase region  $L_1+L_2+\delta$ . Two surfaces of two-phase reactions  $L_1 \rightleftharpoons L_2$  and  $L_2 \rightleftharpoons L_1$  are appeared and both these surfaces have a common tie-line (between the  $L_1(L_2)$  and  $\delta$  phases). One, two or three surfaces of two-phase reaction can appear at the deformation of phase region  $L_1+L_2+\delta$ .

For T-x-y diagram with invariant syntectic equilibrium with binary compound ( $\gamma$ ) the effect of change of three-phase reaction takes place in phase region  $L_1+L_2+C$  but not  $L_1+L_2+\gamma$ . In this case two surfaces of two-phase reaction adjoining to tie-line (between the  $L_1(L_2)$  and C phases) were found.

**Keywords:** *immiscibility gap, phase diagram, computer model*



## **Session 12.**

# **Rheology of Biological Systems**

## Rheology of Fresh Blood

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The blood circulatory system is a case of solid-liquid two-phase flow with *hematocrit* as the solid phase and *plasma* as the liquid phase. Hematocrit is mainly composed of erythrocytes (or red blood cells, RBCs), leucocytes and thrombocytes (platelets). Plasma, is a solution of proteins (albumin, globulin and fibrinogen), coagulation factors and electrolytes. Rheology of blood is extremely complicated for reasons: (1) aggregation and redispersion behavior of RBCs and other cells brought about by shear rate and interaction with other non-cellular components; (2) the viscoelasticity of the RBCs, as the main component of hematocrit; (3) the viscoelasticity of the plasma due to the presence of proteins (which are complex polymers), proportional with their concentration.

Blood samples obtained by venipuncture are subjected to rheological analysis in a controlled stress/strain rheometer using a cone/plate sensor, in this work. Tests are performed at normal body temperature of 37°C and are conducted both with fresh blood and with blood mixed with EDTA (ethylenediamine tetracetic acid, used to prevent blood coagulation). The clear serum remaining above the fresh blood samples after sedimentation and plasma remaining above the blood mixed with EDTA is separated and subjected to the same tests. Viscosities of fresh blood together with its serum and blood taken into EDTA and its plasma are investigated over a range of shear rates  $10^{-4} \leq \dot{\gamma} \leq 10^{+4}$  for full evaluation of blood rheology under different conditions. Creep and recovery tests, first normal stress differences ( $N_1$ ), and dynamic tests are also performed to elucidate the viscoelastic properties of blood and its components.

Blood behaves as a concentrated suspension because of a hematocrit level of 32.7%v/v and higher concentration of globulin and fibrinogen molecules in blood samples. Hence, only fresh blood approaches a constant value of zero-shear viscosity at low shear rates ( $\dot{\gamma} \leq 10^{-3} \text{ s}^{-1}$ ) and the Newtonian viscosity at higher shear rates ( $\dot{\gamma} \geq 10^2 \text{ s}^{-1}$ ). All other samples show only shear thinning behavior and the constant viscosities (5-6mPa.s) at high shear rates. The effect of EDTA does not change the shape of the viscosity curve but creates an order of magnitude decrease because of the decrease in the solid fraction in blood with destruction of structure of platelets with EDTA. Plasma and serum act more like polymeric solutions. Also, various models are used to try to predict the shear behavior of blood. Dynamic tests show that storage modulus of elasticity ( $G'$ ) is higher than the viscous modulus ( $G''$ ) in fresh blood, indicating the dominance of elastic forces. However, serum acts more like a liquid with  $G''$  greater than  $G'$ . In addition,  $N_1$  of fresh blood is measured to be almost equal to that of serum. This signifies that the suspending medium also has viscoelastic properties.

**Keywords:** fresh blood, rheology, viscoelasticity, serum, plasma, concentrated suspensions, non-Newtonian fluids

## **Rheology and Porosity Effect on the Proliferation of Pre-osteoblasts on Zirconia Ceramics**

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Among bioceramics, zirconia (ZrO<sub>2</sub>) exhibits good biocompatibility and outstanding mechanical properties and is suitable for use in medicine. Structural features such as porosity and pore interconnectivity are believed to be crucial for cell growth and osseointegration of implants. The objective of the present work was to compare the adhesion and proliferation of pre-osteoblasts on zirconia sintered ceramics of different porosities.

Yttria stabilized zirconia (YSZ) of different degrees of porosity (19%, 32%, 50%) with hydroxyapatite (HA) coating on porous surfaces were fabricated by sintering powders obtained by plasma-spray pyrolysis. HA coating were obtained by infiltration of salts water solution via porous skeleton. Mechanical properties were characterized by compressing strength tests. Porosity was determined by the Archimedes method and pore size distribution was measured by scanning electron microscopy. Cell metabolic activity and proliferation were assessed up to two weeks based on the redox reactions in living cells, by the PrestoBlue™ assay, as well as by fluorescence microscopy.

The Young's modulus of the porous YSZ ceramics was determined to be between 2-7 GPa, suggesting their feasibility for load-bearing applications. Pore diameter varied between 0.1-75μm with mean-size 3-15μm depending on porosity. The metabolic activity of pre-osteoblasts on the high porosity (50%) zirconia surface was significantly higher (about 2-fold) than on the lower porosity surfaces. Higher porosity was found critical for cell proliferation, whereas the presence of HA in the low porosity surfaces had no effect - increasing the porosity to 50% resulted in a pronounced stronger effect on adhesion, with excellent cell spreading and proliferation leading to complete surface and pore coverage.

The data indicate that in high porosity zirconia, pores provide a crucial advantage for cell ingrowth, and render an otherwise bioinert material into a bioactive macroporous scaffold for bone tissue growth.

## **Numerical Simulation of Rheological, Chemical and Hydromechanical Processes of Thrombolysis**

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The aim of the work was to build a complex three-dimensional model of rheological, hydro- and chemical processes in a deformable clot, which occur during the thrombolysis. The clot is considered a porous medium with elastic or elastic-viscous rheology. Fibrin of the clot is described as stationary solid phase, plasminogen, plasmin and plasminogen-activators are described as dissolved fluid phases. A numerical solution based on the obtained model is developed. The aim of the numerical simulation was to investigate dynamics of the thrombolysis, the transport of components in the fluid and solid phases, the influence of clot characteristics on the process of the lysis. Obtained results show good correlation with works of predecessors and demonstrate significant influence of rheological properties of the clot (fibrin) on the process of the lysis. Three-dimensional picture of clot dissolution is obtained. It is shown that the bigger elasticity causes higher rate of clot destruction. The comparison of results for the elastic and elastic-viscous rheologies of the clot is performed. The dissolution of the clot goes even faster in the case of the elastic-viscous rheology. An assumption about the acceleration of the dissolution during the process of the lysis can be made. The numerical experiment shows that the clot is dissolved in approximately 40 minutes, and the speed of the lysis is increasing during the process.

**Keywords:** *thrombolysis, rheology of porous media, numerical simulation*

## **Rheological regional properties of brain tissue studied under cyclics creep/recovery shear stresses**

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The rheological properties of brain tissue were studied by repeated creep-recovery shear tests under static conditions for different regions, Corpus callosum CC, Thalamus Th and Corona radiata CR. Non-linear viscoelastic model was also proposed to characterize the transient/steady states of shear creep results. The creep/recovery data were determined by a control stress rheometer in parallel plate configuration. From the creep-recovery data it was obvious that the brain tissues show high regional anisotropy. However, the both samples exhibit fluid viscoelastic properties in the first shear stress cycle of 100 Pa, while this behavior evolutes to solid viscoelastic with cyclic effect.

**Keywords :** *Brain tissue mechanics, Corpus callosum, Creep-recovery, Viscoelastic, Thalamus, Corona radiata*

**Session 13.**  
**Rheology of Construction Materials and  
Concretes**

## **Study of the rheological properties of cement mortar mixture with calcined clay**

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The effect of calcined clay (CC) used as partial replacements for cement on the rheological properties of cement mortar without superplasticizer by using a rotational viscometer and the flow test was investigated. Various proportions of 10%, 15% and 20% calcined clay (CC) were individually used as partial replacement for cement in 0,50 to 0,70 water/cement (w/c) ratio cement mortar mixtures. The rheological properties of cement mortar mixtures with calcined clay (CC) were compared to cement mortar mixtures containing respectively 10% fly ash and silica fume.

The yield stress and the plastic viscosity decreased with replacing cement with fly ash (FA). In the case of cement-silica fume (SF) system, yield stress and plastic viscosity steeply increased. For cement mortar mixture with fly ash (FA) and without partial replacement, the rheological properties improved much more than cement mortar mixture with respectively silica fume (SF) and calcined clay (CC).

The factors influencing this phenomenon are the different type of structure and the effect of adsorbed water on the surface of the mineral admixtures.

**Keywords:** *Rheology, Calcined clay, replacement, flow test, yield stress, plastic viscosity, silica fume, fly ash, w/c-ratio, particle structure*

## **A Primary Study on the Creep Properties of Eucalypts\***

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The paper tested the flexural creep curves of Eucalypts under different loads, different moisture contents and different temperature levels in 5 hours, and run the conclusion as follows:

① The creep curves of Eucalypts under different loads abide by the similar rule, which will be raise with the addition of loads.

② To heighten the moisture contents of Eucalypts can increase the creep, and creep rupture will be easy happened under higher degree of moisture capacity.

③ The temperature is factor that influence the creep of Eucalypts distinctly, and the decelerating creep will come to clear with the increase of temperature.

④ Burger's rheological model can be used to simulate the early stage of creep of Eucalyptus.

**Key words:** *Eucalyptus; flexural creep; load; moisture content; temperature*

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## **Study of the rheological properties of cement mortar mixture with calcined clay**

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The effect of calcined clay (CC) used as partial replacements for cement on the rheological properties of cement mortar without superplasticizer by using a rotational viscometer and the flow test was investigated. Various proportions of 10%, 15% and 20% calcined clay (CC) were individually used as partial replacement for cement in 0,50 to 0,70 water/cement (w/c) ratio cement mortar mixtures. The rheological properties of cement mortar mixtures with calcined clay (CC) were compared to cement mortar mixtures containing respectively 10% fly ash and silica fume.

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**Keywords:** *Rheology, Calcined clay, replacement, flow test, yield stress, plastic viscosity, silica fume, fly ash, w/c-ratio, particle structure*

## **Mineral admixture influence on thixotropic behaviour of light cement**

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This paper deals with the effect of Portland cement and gypsum–Portland cement ratios on the rheological properties of light cement, specially the thixotropic behaviour. The results indicate that the setting time of these paste decreases with the increase of gypsum content in the mixture, by increasing early strength and stabilizing viscosity without changing thickening time.

The porosity of blended gypsum binders ranges from 12% to 37%. Their water absorption is high, reaching 27% in the blends with a greater proportion of gypsum. The gypsum–Portland cement blends themselves possess good water resistance, which is further enhanced by the addition of bentonite and salt. These blends give excellent properties retention after aging in water at 20°C for 95 days. Their good resistance to water decreases as the gypsum content in the mixture is raised. However, the strength loss for the gypsum–Portland blends is generally less than that observed for the gypsum binder. Structural and mechanical characterizations have been also studied such as compressive strength, durability, expansion etc.

**Keywords:** *Portland cement, gypsum, rheology, thixotropy, salt, mechanical, durability.*

## **Natural materials to enhance rheological and mechanical properties of light cement for oil wells**

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One of the most serious problems encountered when cementing casing in a well is the failure of casing and cement formation bond. This is a major problem from the stand point of allowing migration of fluids from one zone to another. Also poor bonding can result in large losses of reservoir fluids and premature reservoir depletion. This work concerns the elaboration of well drilling cement with local materials for unconsolidated formation in the south of Algeria. This paper describes the properties and uses of cementitious mixture containing different ratio by weight of cement of natural adding materials such as bentonite, salt and diatomaceous earth. Rheological behavior have been investigated specially thixotropic characteristics to reach optimum hydraulic parameters while cementing. Structural characterization have been also studied by several methods such as DRX and SEM microscopy helping understanding the mechanical parameters of set materials and finding correlations with the compressive strength expansion and free water values etc... The results indicate that early strength is increasing and viscosity stabilizes without changing thickening time on the physical, mechanical and durability properties of PORTLAND cement-diatomaceous earth blends with increasing of setting time at different dosage. Key words : cementing, oil well, local materials adding, cement, salt, bentonite, diatomaceous earth, rheological behavior, thixotropy, structural, mechanical parameters, durability.

## **Influence of mineral aggregates to the rheological properties of concrete mixture**

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The aim of this research was to determine how the changing of concrete mixture constituents: fine and coarse aggregate concentration as well as the amount of fine particles, not exceeding 0.25 mm, influence concrete mixture's rheological properties. Firstly, inner-concentration of fine aggregate (sand, fraction of 0/1 and 0/4) was changed. Secondly, coarse aggregate (gravel, fraction of 4/16) concentration was changed and finally, the amount of fine particles was changed in order to investigate how it influences concrete rheological properties.

Results have shown that by the increase of sand (fraction of 0/1) quantity, the plastic viscosity also increases. On the other hand, yield stress, at the beginning decreases, but eventually – increases. The increase of coarse aggregate quantity acted differently: plastic viscosity and yield stress decreased. Finally, the increase of fine particles quantity decreased the plastic viscosity as well as yield stress of concrete mixture.

**Keywords:** *Yield stress, plastic viscosity, concrete mixture, fine particles, coarse aggregate.*

**Session 14.**  
**Rheology of Emulsions, Suspensions  
and Foams**

## **The Effect of Fructose on Rheological Behavior of Alumina Dispersions**

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Colloidal processing of dispersion usually requires identification of flow characteristics. Many rheological techniques can be used to reveal insight information about the dispersion structure. The use of mono and disaccharides can be used as alternative dispersants for ceramic. The previous investigations explain the viscosity reduction by adsorption of sugar based dispersants which eliminates bound water around particle surface. The aim of this study is to investigate the use of fructose as a dispersant for alumina suspensions dispersions by means of rheological techniques.

Two different alumina powders were used in this investigation. One of them is defined as submicron alumina (supplied from Sumitomo Chemicals Co., Ltd., Japan) and the other is nano alumina (supplied from Alfa Aesar GmbH & Co, Germany). The average particle size of the submicron alumina and nano alumina were about 0.2  $\mu\text{m}$  and about 40 nm, respectively. The suspensions having 10-40 vol% solids were prepared in 1-20 wt% fructose solutions. The rheological measurements were done on a rotational rheometer (Haake Mars II Advanced Rheometer) by using parallel plate measurement apparatus in 35 mm diameter at 20°C. The preshearing was applied for 60 seconds at 50 1/s and waited at rest for 600 seconds before dynamic rheological measurements. The average of three measurements were reported and standard deviation was calculated in 0.05 confidence interval. The flow behavior of the dispersions were determined between shear rates of 0 and 400 1/s. The dynamic experiments prolonged much longer time than the steady shear experiments. The successive stress sweep experiments showed that a significant change in the linear viscoelastic region. The magnitude and the shear stress range of  $G'$  shifted to much higher values due to drying of sample. The presence of a vegetable oil around sample-air contact eliminated the drying problem [2].

The submicron and nano alumina dispersions in fructose solution had shear thinning behavior and were fitted to the Herschel-Bulkley model. The presence of fructose reduced the viscosity of submicron alumina dispersions. However, there was no significant effect of fructose on viscosity of nano alumina dispersions at high solid loadings. The microstructures of the suspensions were investigated by applying dynamic shear rheology tools. The frequency sweep tests showed that the given energy was stored by solid part of the dispersions. It was observed that fructose concentration has also additional effect on the elastic moduli ( $G'$ ) of the dispersion. At 40 vol% solids loading, the  $G'$  was independent of angular frequency. The nano alumina dispersion has more solid like and elastic structure than submicron alumina dispersions.

As a conclusion, nonionic small molecules like fructose can be used to regulate rheological behavior of ceramic powder dispersions. These environmentally friendly molecules may become new dispersants for the ceramic powder dispersions. However, the performance of sugar based dispersants is quite dependent on the powder characteristics such as particle size and surface area.

**Keywords:** Nano powders, Alumina, Fructose, Viscosity, Rheology, Dynamic Shear Measurement

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## **Rheology & Thermal Stability of Ethoxylated Surfactants and there interaction with Polymers used for Enhanced Oil Recovery.**

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The objective of my study is to study the interactions between the ethoxylated Surfactants and polymers. Polymers and surfactants are used in the Enhanced Oil Recovery to increase the sweep efficiency by increasing the viscosity of the water and decreasing the Interfacial Tension between water and oil respectively. Research will be conducted on different formulations of surfactant and polymers and vary different parameters. With the help of these research results we shall be able to optimize the particular formulation of SP system for specific conditions. Effect of different types of surfactants and polymer blends on rheology and thermal stability will be observed and so the best system will be recommended. Salinity and salts affect the viscoelastic properties of SP system so the optimum salinity which gives the best results will be preferred. Effects of temperature, Pressure and Shear rate will be studied and recorded for further use in future.

After all these results analyzed and logged, one will be able to use the best SP system for particular application. As carbonate reservoirs which contain 60% of oil in the world, are still to be treated. After this research, it will help to select the best system to recover maximum oil from these reservoirs and also from others as it will help to understand which system will perform best under specific conditions.

**Keywords:** *Surfactant, Chemical Enhanced Oil Recovery, Rheology, Thermal Stability*

## **Rheological Properties of Liquid and Particle Stabilised Liquid Foam**

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In Earth-Pressure-Balance (EPB) tunnelling, the surface settlements may be prevented by using excavated ground as face support medium, controlling ground movements and ground water flow into the excavation chamber. The excavated ground (soil) does not exhibit suitable conditions to be used as support medium. A suitable condition for the excavated soil can be achieved by mixing conditioning agents that are mainly foams. To ensure a homogeneous pressure transfer onto the tunnel face, the conditioned soil, i.e. the soil-foam mixture, requires a certain effective viscosity. This is also important for an effective transport through the excavation chamber. Understanding the rheological properties of the (added) foam and the foam-soil (foam-particle) mixture, and its influence on the soil is not possible without advanced experimental investigations. Therefore, various rheological experiments such as flow curve tests, oscillation tests, and further experiment to determine the effective yield stress are performed. In detail, (homogeneous) rheological experiments are performed by using plate-plate and cone-plate geometries with modified, i.e. rough surfaces in order to prevent slip effects. Since the microstructure of the foam accounts for the effective rheological properties of the complex fluid, the size, shape and distribution of the foam and particle-laden foam is characterized in detail by imaging techniques. In order to perform the above mentioned experiments, polymer-stabilised shaving foam seems to be a good choice in laboratory tests due to its time stability and easy accessibility. On the other hand, glass beads (of different diameter and volume fractions, i.e. specific surface areas) are used to understand the effective material behavior of foam-particle mixtures. The experimental results are compared with rheological yield-stress models of Herschel Bulkley type.

**Keywords:** EPB tunneling, soil conditioning, foam, rheological experiments, Herschel Bulkley model



## **Rheological behavior of Pickering emulsions stabilized by phyllosilicates**

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Surfactant-free emulsions stabilized by solid particles, also called Pickering emulsions, have been evaluated in this study. The development of an elastic film at the oil-water interface following the adsorption of colloidal particles helps to promote Pickering emulsion stability<sup>1</sup>. The purpose of this investigation was to examine the formulation, droplet size distribution and stability of Pickering emulsions using natural model phyllosilicates: kaolin, palygorskite, talc and halloysite. Formulation of Pickering emulsions was optimized in order to prepare oil-in-water (o/w) and water-in-oil (w/o) emulsions stabilized with different amounts of the aforementioned phyllosilicates and dodecane as an oil phase.. The stability of the emulsions was first evaluated by bottle tests and by direct observations with an optical microscope. Thereafter, the impact of phyllosilicates on the dynamic rheological parameters for stable emulsions had been investigated by measuring the elastic and loss moduli  $G'$  and  $G''$ , and complex modulus,  $G^*$ . The cohesive energy, corresponding to the dissipated energy per volume unit when the network is broken could be obtained for these emulsions.

Results showed that stable and homogeneous emulsions obtained with kaolin and halloysite exhibited smaller droplet size than those obtained when using attapulgite or talc. These results were confirmed using the rheological stability criteria, that for highly stable emulsions:  $G' > G''$  and both  $G'$  and  $G''$  are independent of frequency. It was concluded that highly dense, stable and concentrated elastic structure of w/o Pickering emulsion was obtained when mixing phyllosilicates (15 wt%) in the aqueous phase. Moreover, talc Pickering emulsion exhibited the highest cohesive energy that can be explained by internal structure of emulsions according to the degree of packing of emulsion's droplets.

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**Keywords:** *rheology, Pickering emulsions, phyllosilicates, stability.*

## **Investigation of rheological properties of porcelain suspensions**

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The rheological behaviour of porcelain raw material suspensions were studied by authors under different technological conditions so that better control can be achieved during the slip casting processes. Factors, like slip temperature, mixture density and shear rates have measurable effects on rheological properties of the ceramic suspensions.

Slip casting is a very time-intensive process, and during slip casting casting rate can be enhanced by raising the temperature of the slip and the mould. Thus in this present work viscosity and thixotropy of the slurry were investigated at different temperatures ranging from 25°C to 40°C. Datas on the rheological properties of the slip were obtained at fixed and varying shear rates, different suspension densities and temperatures.

In their work the authors tried to give a rheo-mechanical model of porcelain raw material suspensions used in "Álföld" porcelain plant.

**Keywords:** *porcelain, rheology, thixotropy, viscosity, slip casting, slurry, suspension*

## **Influence of the nature and the amount of surfactant on the rheological behavior of model phyllosilicate suspensions**

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Phyllosilicates are abundant raw materials offering a wide range of applications, from traditional ceramics (e.g. tableware), to functional high-added value ceramics (e.g. filtration membranes). If individual kaolin and smectite aqueous suspensions have been extensively investigated, further studies need to be performed using other clay minerals, which are process and applications relevant regarding clay-based ceramics. In this way, the present work aims at studying the rheological behavior of single model phyllosilicate suspensions and at comparing the effect of different surfactants on their dispersion/deflocculation.

High purity commercial clays were used as model phyllosilicates, namely: BIP kaolin, NZCC halloysite, ABM illite and Yellowstone 140 talc. Characteristics of these raw materials, such as particle size distribution, specific area and density were determined. Phyllosilicate aqueous suspensions with solid content of 2, 4, 8 and 16 mass% were characterized using a stress-controlled rheometer in the flow mode.

Parameters controlling the dispersion of the previous phyllosilicate suspensions, such as the nature and the amount of the dispersant were investigated by considering above all their rheological behavior, zeta potential and particle size distribution. More precisely, the rheological parameters were determined using Herschel-Bulkley relation. As expected, results indicated a shear-thinning behavior for the studied phyllosilicate suspensions. The deflocculation action of the different surfactants was noted through the decrease of both yield stress and consistency. Correlation with zeta potential values allowed determining the optimal deflocculation/dispersion conditions.

Concerning talc suspensions, the studied surfactants did not lead to significant trend regarding dispersion or deflocculation. For kaolin, halloysite and illite suspensions, polyanions seemed to be the most suitable additives for developing shear-thinning and low yield stress suspensions.

**Keywords:** *phyllosilicate; dispersion; rheology; deflocculation; particle interaction*

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## **Flow of High Internal Phase Ratio Emulsions through Converging Nozzles**

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High internal phase ratio emulsions (HIPRE) have structured continuous phases that cause these emulsions to exhibit different rheological behavior under different flow conditions. The continuous phase is usually a polymer or a surfactant in solution. Surfactants with a linear hydrophobic chain and a small hydrophilic group with approximately the same cross sectional area as the hydrocarbon chain form lyotropic liquid crystals. The van der Waals forces among the hydrocarbon chains and the hydrogen bonds among the hydrophilic groups of the closed packed surfactant molecules at the interface resist stretching under elongational flow conditions. Extensional viscosity ( $\eta_e$ ), is an indication of resistance of the secondary bonds toward stretching. Extensional viscosity arises on the increase of velocity when the cross sectional area of flow is reduced in the flow direction. Velocity of the molecules with negligible interaction forces increase independently in the case of Newtonian fluids. In the case of complex fluids, elongational flow causes the development of normal stresses within the fluid.

The volumetric ratio of the internal phase of an emulsion can be increased above 74% when lyotropic liquid crystals are used as surfactants. The external phase and the surfactants are then present as a thin film surrounding the polyhedral internal phase and in the Plateau borders at the corners. Mechanical energy input, as in the case of stirring of the emulsion is converted into surface energy, reducing the size of the drops, increasing the interfacial area and reducing the amount of bulk liquid in the Plateau borders. The yield stress of the emulsion increases with the extent of the interfacial area. Elongation of the bulk phase emulsion will readily be transferred to the surfactant bilayers at the interface and the emulsion will acquire viscoelastic properties. The ratio of the extensional viscosity to the shear viscosity will exceed the value  $\eta_e/\eta_s = 3$  given by the Trouton's rule.

The shear and extensional viscosities effective in flow through converging pipes is investigated for the case of 94% W/O HIPRE as a function of prestirring rate before flow. The internal phase consisted of 1% sorbitol solution, and the external phase, a mixture of mineral oil and polyoxyethylene (2) oleyl ether (Brij 92) at a 1/1 volumetric ratio. The emulsions were formed at 60 °C with stirring at a rate of 400 rpm, the minimum rate for the formation of stable HIPRE. Before the flow experiments the emulsions were stirred again at 400, 600, 800 or 1000 rpm rates for 20 minutes. The viscosities of the emulsions were measured with Haake rheometer, using the bob and cup attachment. The flow set-up consisted of a storage tank under nitrogen atmosphere, pipes leading to the converging pipe and pressure gages. The flow rate was adjusted with the pressure of the nitrogen gas in the storage tank. The outlet to inlet ratio of the diameters of the converging pipe was,  $D_o/D_i = 9 / 21$ , and the calculated velocity ratio for a Herschel-Bulkley model fluid would be  $u_o/u_i = 5.44$ .

Elongational to shear  $\eta_e/\eta_s$  viscosity ratios exceeding Trouton's rule could be obtained only in the case of 600 rpm prestirring rate. The low values at 800 and 1000 rpm prestirring rates were attributed to the rupture of the bilayers forming the external phase under high wall shear stresses. Slip flow prevalent under these conditions were also confirmed by experiments in glass tubes. The experimental results obtained in this work are evaluated with the rheological models obtained by viscosity measurements and drop size distributions.

**Keywords:** *high internal phase ratio emulsions, extensional viscosity, shear viscosity, lyotropic liquid crystals, converging pipes, elongational flow.*

## **A Rheological Approach to Estimate floc Structure in Concentrated and Flocculated Suspensions**

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Concentrated suspensions, where microparticles are dispersed in a liquid phase, are widely used in industries, ranging from concrete in construction industry to the precision surface chemistry industry, and it is important to predict the complex rheological behavior of such suspensions. For the case of flocs, particles flocculate to fractals, and maintain their fractal properties even when they are fractured by shearing and re-flocculation<sup>1)</sup>, many predictions of fluid properties based on fractal aggregate models have been proposed<sup>2)3)</sup>. This study examines the effect of increases in shear rate and volume fraction on the structure of a fractal floc by using a rheology model to compute the fractal dimension of the fractal floc in coagulation concentrated suspensions under various shear rates and volume fractions.

Mills expressed the viscosity of coagulation concentrated suspensions as Equation (1), using floc effective volume fractions instead of particle volume fractions<sup>2)</sup>:

$$\eta_r = \frac{1 - \phi_{eff}}{(1 - \phi_{eff} / \phi^*)^2}, \quad (1)$$

where  $\eta_r$ ,  $\phi_{eff}$  and  $\phi^*$  represent the relative viscosity (-), the floc effective volume fractions(-), and the random packing volume fractions(-), respectively.

This study assumed the following relationship between the effective volume fraction and shear stress by considering the balance with shear stress and interparticle forces<sup>3)4)5)</sup>;

$$\phi_{eff} / \phi \propto \tau^{-\frac{(3-D)}{(4-D)}} \quad (2)$$

where  $\phi$ ,  $\tau$  and  $D$  represent the solid particle volume fraction(-), shearing stress (Pa) and fractal dimension(-), respectively.

The model was applied to the experimental data of mono- and poly-dispersed suspensions in the present and previous studies. This study used suspended silica particles of average radius 2.4 $\mu$ m and the narrow particle size distribution of 1.06 – 20.0 $\mu$ m in a 1.0M NaNO<sub>3</sub> solution for 0.05 of change in the volume fraction from 0.15 to 0.55 and also used ordinary portland cement paste for 0.05 of change in the volume fraction from 0.20 to 0.50.

The result showed that the fractal dimension of an aggregate in a sheared suspension increases with volume fraction rather than with shear rate,  $\dot{\gamma}$ , or random packing fraction,  $\phi^*$ . The estimated values were approximated by eq.(3) as a function of volume fraction.

$$D = 3.00 - 0.84\exp(-6.12\phi) \quad (3)$$

It is suggested that, in particular, the fractal dimension values would increase sharply at volume fractions higher than those when percolation occurs. Pertaining to the effect of the shear rate, this study found that the fractal dimension values vary with the volume fraction at high shear rates although such a variation was not confirmed at low shear rates.

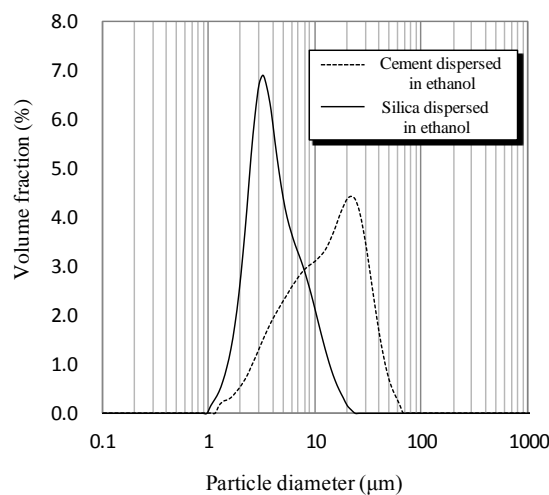


Fig. 1 Particle size distributions for silica particle and cement particle dispersed in ethanol.

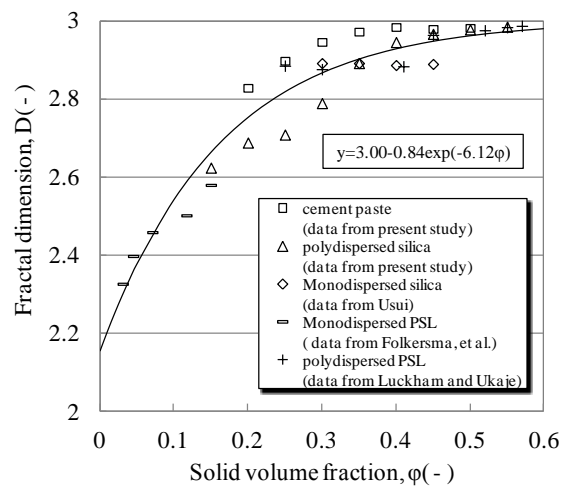


Fig.2 Fractal dimension vs solid volume fraction for flocculated suspensions. Fractal dimension is estimated from flow curves using eq.(1) and eq.(2). The flow curve data is from our experiment and ref.6). 7). 8).

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**Keywords:** suspension, interparticle forces, floc, rheology, fractal dimension, percolation

## **Qualification methods of Al<sub>2</sub>O<sub>3</sub> injection molding raw material**

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Nowadays, the sophisticated ceramic industry and technologies require higher and higher assumptions against quality, volume and yield of end product. [1,2] In the illuminant industry, for producing arc tube parts for high intensity discharge lamps the applied method is the ceramic injection molding. The ceramic arc tube parts are made of high purity alumina powder. By producing ceramic parts, one of the most critical step is to optimizing the injection molding process, determining and controlling the influential machine parameters, which have an effect on the quality of end product. [3] Nevertheless, before we fall to doing the optimization of injection molding process, we need to know the properties of injection molding raw material, because later the molding process will be optimized for this material, to decrease the amount of cracked ceramics. [4]

For producing ceramic arc tube parts (plugs), there are used two different major components for producing injection molding raw material (feedstock): high purity alumina powder as the main component, and an organic paraffin wax as a binder material. It is expressly important to know the material, physical and chemical properties of these components, since mainly these have effect on the homogeneity of feedstock, and therefore on the quality of end product. [5]

In this research, both of the main components and the moldable raw material was investigated by visual, physical, chemical and thermal methods. As most important and main statement, the researchers found that the dynamic viscosity of raw material depends more on the applied temperature, than on the deformation speed gradient. In addition, it was found, that the grain size distribution of alumina powder and the amount of binder material have a huge effect on the dynamic viscosity of molding material.

Applied analytical methods were laser granulometry, sieve analysis, scanning electron microscopy, differential thermal analysis, specific surface area analysis and rheology analysis.

**Keywords:** *alumina powder, paraffin wax, ceramic injection molding, laser granulometry, scanning electron microscopy, thermal analysis, rheology analysis*

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## **RHEOLOGICAL CHARACTERIZATION OF CEMENT OIL SUSPENSIONS**

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This study is a contribution to the study of the rheological behavior of cement suspensions. An oil well is drilled, cased, cemented and set completion. The well drilling is done in several phases (different diameters) to isolate problems (land subsidence fragile and poorly consolidated formations came water, loss of movement in the porous and permeable formations,...), thus, it should go down a casing and cementing to work safely. The cement is injected into the annulus (space between the casing and the formation down) deep formations in order to isolate the aquifers essentially layers licks and also isolate formations important differential pressure ... (incompatible training ) in deep oil well. The materials studied were chosen to meet the requirements and problems encountered in real applications in the oil field (Casing cementing wells), so it makes use of oil hydraulic binder "G". The hydraulic binder "G" is a cement based standardized API (American Petroleum Institute), it is expected to great depths and is compatible with various adjuvants and can be used with accelerators or retarders to cover a wide range of pressures and temperature. This systematic study of rheological properties of cement grout Class "G" would propose a formulation of grout compatible with the surrounding environment, with optimal efficiency and costs under control. Keywords: Oil drilling, cement, rheology.



## **Low temperature dolomitceramics with glass waste additive**

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Composite materials - from low melting clay, dolomite and glass powder in different ratios were modelled by firing the mixtures at different temperatures in interval 600 – 800° C. Both mechanical and physical properties were studied of the sample series after firing and after 7 days of hydration in a dry and wet state. The influence of the amount of added glass powder was evaluated. Pore size distribution measured by mercury porosimetry as well the mineralogical composition – changes of crystalline phases after firing and accordingly after hydration was measured by X-ray diffraction (XRD) for the samples with optimal mechanical properties.

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**Keywords:** *clay, dolomite, waste glass, modelling, composite material, ceramic*

**Session 15.**  
**Rheology of Ferrofluids and Magneto-  
rheological Materials**

## **Mechanical Loss in Multiferroic Materials at High Frequencies: Friction and the Evolution of Ferroelastic Microstructures**

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Energy absorption in multiferroic materials stems typically from strain relaxation which can be strong even when no extrinsic defects exist in the material. Our computer simulations of a simple two-dimensional model on a generic, proper ferroelastic material have identified the dissipative mechanisms associated with the dynamical motion as 1.) Advance and retraction of needle-shaped twin domains and, 2.) Movement of kinks inside twin boundaries. Both movements involve friction losses. The friction loss in needle movements is almost frequency independent. The loss occurs mainly during the pinning and de-pinning of the needle at another twin wall. The dynamical response involves a retardation of the needle movement relative to the applied force. This phase angle is strongly frequency dependent, as observed experimentally in Resonance Ultrasonic Spectroscopy. Freely traveling kinks propagate with a constant speed, any higher forcing leads to stronger phonon scattering but does not increase the speed. No defects are involved in these mechanisms.

**Keywords:** *dynamical energy dissipation; mechanical spectroscopy; needle domains and kinks; intrinsic pinning and depinning*

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Z. Zhao, X. Ding, T. Lookman, J. Sun, and E.K.H. Salje, Mechanical loss in multiferroic materials at high frequencies: friction and the evolution of ferroelastic microstructures, *Advanced Materials*, 2013, DOI: 10.1002/adma.201300655

## **Rheology of novel magnetic fiber suspensions**

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Rheological properties are of importance for soft matter and, in particular, for colloidal suspensions. When the suspended particles are made of magnetic materials, the rheological properties of the suspensions can be tuned, in a fraction of second and in a reversible way, by application of a magnetic field. This phenomenon is known as magnetorheological effect and the suspensions displaying it are commonly called magnetorheological suspensions. The adjustability of the rheological properties of the magnetorheological suspensions is the base for many potential technological and biomedical applications of these smart materials. During the last few decades, material scientists have been trying to prepare magnetorheological suspensions that combine the required colloidal stability and a strong enough magnetorheological effect to make viable the commercial use of these materials in applications. In this work we show recent results that prove that the use of magnetic fibers as dispersed particles allows the preparation of magnetorheological suspensions that combine these two properties. Colloidal stability of suspensions of magnetic fibers is directly related to the fiber-like shape of the dispersed particles. This shape implies that particle concentrations of practical interest are close to the onset of the concentrated isotropic regime, in which the fibers begin to have difficulty packing isotropically and, consequently, phase separation is prevented. With respect to the magnetorheological effect, both steady-state and dynamic rheological measurements indicate that when magnetic fibers are used this effect is considerably enhanced, compared with that exhibited by conventional magnetorheological suspensions (based on spherical particles). We show that this enhancement is likely to be a result of the existence of interfiber friction with strength dependent on the intensity of the applied magnetic field. In addition to these results, suspensions of magnetic fibers present some unusual rheological behaviors. In particular, curves of the shear stress as a function of the shear rate have a decreasing branch at low shear rate, that is, a region of negative differential viscosity. From the point of view of basic science, these unconventional rheological behaviors are of interest and are likely to be found in other materials. With respect to applied science, suspensions of magnetic fibers are more appropriate for applications in magnetorheological devices than suspensions of magnetic spheres because of their enhanced colloidal stability and magnetorheological effect.

## Modelling of a magnetorheological fluid under shear deformation

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In recent years the interest in materials with specific adjustable properties has increased due to higher requirements on the material performance. Therefore, the project ECEMP (European Centre for Emerging Materials and Processes Dresden) has been created. In the subproject ECEMP-B4 a smart composite made of a magnetorheological fluid (MRF) embedded in a polymeric matrix material is considered. Its material behaviour is to be predicted by a multiscale modelling technique which requires the knowledge of the material behaviour of each constituent. Here the behaviour of the MRF under shear deformation is investigated.

The experimental characterisation was done by the Chair of Magnetofluidynamics (ODENBACH, Faculty of Mechanical Science and Engineering, TU Dresden). The MRF was self-made by mixing 40 vol% iron particles with a main diameter of 5  $\mu\text{m}$  from BASF into the carrier liquid Motul 300V. Its flow curve  $\tau(\dot{\gamma})$ , the storage  $G'(\dot{\gamma})$  and loss modulus  $G''(\dot{\gamma})$  as function of the shear amplitude  $\dot{\gamma}$  were measured without a magnetic field as well as for  $|\vec{B}_1| = 50\text{ mT}$ ,  $|\vec{B}_2| = 150\text{ mT}$  and  $|\vec{B}_3| = 300\text{ mT}$ .

In this contribution the MRF is treated as homogeneous continuum and is modelled in a phenomenological way. Its constitutive equations are motivated by the nonlinear flow curve which also shows a dynamic yield stress, the decreasing character of the storage modulus and the pronounced maximum of the loss modulus. Thus the material behaviour of the MRF is modelled by a parallel connection of a yield stress fluid and a nonlinear viscoelastic MAXWELL element with a nonlinear shear-rate-dependent ansatz  $\eta(\dot{\gamma})$  following a reduced approach of the CROSS model [1]. The model prediction of the storage and loss modulus is calculated with respect to large amplitude oscillatory shear (LAOS). Characteristic points are identified and connected to the material parameters so that they can be determined by a physically motivated identification procedure.

- [1] J. Claracq, J. Sarrazin, J.-P. Montfort, *Rheologica Acta*, 43, p. 38, 2004.

**Keywords:** Magnetorheological fluid, Yield stress fluid, Nonlinear Viscoelasticity, CROSS model, Large amplitude oscillatory shear

**Acknowledgments:** This project (ECEMP-B4; Nr.: 100111530) is funded by the European Union and the Free State of Saxony.

## **Magnetorheology of the carbonyl iron/ZnO rod-like microparticle suspensions**

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Magnetorheological (MR) suspensions are generally two-phase systems consisting of magnetic particles and silicone oil. Upon application of an external magnetic field particles become magnetized and form chain-like internal structures, resulting in the change of the rheological behaviour of whole suspension. Viscosity can increase in several orders of magnitude and then return to its original value when magnetic field is replaced. Thus, the viscosity of such suspensions can be controlled using various magnetic field strengths. Reversible change of viscosity can find application in damping systems or torque transducers.

This study is aimed on the preparation of the novel magnetic particles assembled from carbonyl iron (CI) core and ZnO rod-like shell. The hybrid CI/ZnO microparticles were prepared via two-step synthesis. Firstly, the CI particles were seeded with ZnO crystal nuclei and further with the help of hydrothermal synthesis the rod-like structures were formed. Prepared particles were characterized using SEM technique as well as XRD analysis. Further, the magnetic properties of both bare CI particles and CI/ZnO rod-like ones were investigated using vibration magnetization saturation method. The rheological properties of the suspension consisting of bare CI and CI/ZnO rod-like were elucidated in the absence as well as in the presence of the applied external magnetic field strength. The presented core-shell material based suspension exhibited similar MR efficiency in comparison to the suspension based on bare CI particles. On the other hand, the thermal stability of the particles was considerably enhanced. Thus, the main aim of this contribution will be focused on the magnetorheological behaviour of novel CI/ZnO rod-like particles, at broad temperature range.

**Keywords:** *magnetorheology, carbonyl iron, ZnO rod-like particles, magnetic properties, suspensions, rheological properties*

### **Acknowledgement**

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## **Determination of the liquid steel viscosity curves using a high temperature rheometer**

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Rheological measurements of high melting temperature materials require specialist equipment, which allow to obtain high temperature inside. This situation take place in case of iron alloys. In literature there is lack of information about rheological properties of liquid iron solutions. This paper is intended to supplement the data in this regard; included in the study of iron solutions rheological properties: five chemical composition with carbon amount in the range between 0.39 – 0.89 %, while used shear rate in the range between 40-180 s<sup>-1</sup>. Obtained viscosity values are set in the range between 0.002-0.018 Pa·s. The obtained results could be used for verification of the model which calculate the parameters of liquid iron alloys during casting, in which the viscosity is one of the mane parameter.

**Keywords:** *viscosity, liquid steel, rheology, rheometer*

## **Magnetorheological fluids with two-component dispersed phase**

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In fluid media containing non-colloid magnetic particles as a dispersed phase, effective viscosity can increase by some orders under the magnetic field influence because of structurization of particles. Such media are called magnetorheological fluids (MRF). Ability of MRF to be structured in the magnetic field is defined substantially by their magnetic properties – magnetic permeability, saturation magnetization, remnant magnetization, coercive force.

MRF containing the complex dispersed phase consisting of two types of particles in various ratios are investigated. A series of compositions with volume concentration of the dispersed phase near maximum possible (about 50 %) is studied. These compositions contained as one component of a dispersed phase particles of soft magnetic material – carbonyl iron, as the second – particles of hard magnetic material (chrome dioxide  $\text{CrO}_2$  or iron gamma-oxide  $\gamma\text{-Fe}_2\text{O}_3$ ) or non-magnetic particles of aerosil  $\text{SiO}_2$ .

Flow curves in the mode of continuous deformation are received, and also values of static yield stress  $\tau_0$  are defined by the procedure of shear stress growth at various induction of the magnetic field  $B$  in the range up to 1 T by means of rheometer Physica MCR 301 by Anton Paar with using of a measuring cell of parallel plate type with diameter of 20 mm. Magnetic properties of MRF are defined by the standard technique of measurement of magnetization with two Hall sensors. Magnetization curves are received in the range of magnetic field strength up to 450 kA/m.

Character of influence of a material of particles of the second type on rheological and magnetic properties of MRF are defined. MRF, containing 42 vol. % of carbonyl iron and  $\text{SiO}_2$ , having the least values of saturation magnetization and shear stress, has the largest the relative increment of shear stress (more by 700 times in the magnetic field with  $B=0.7$  T at shear rate  $30 \text{ s}^{-1}$ ), which allows to expand control range of controlled hydraulic systems. MRF, containing 48 vol. % of carbonyl iron and  $\text{CrO}_2$ , having the most high values of saturation magnetization, has the relative increment of yield stress is one order lower than for all other MRF.

**Keywords:** *magnetorheological fluid, two-component dispersed phase, shear stress, magnetization, rheological properties, control range*



**Session 16.**  
**Rheology of Fluid Interfaces and Modeling**

## **Structure and interfacial rheology of 2-D suspensions of oppositely charged particles**

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Monolayers of colloidal particles at fluid - fluid interfaces form an important class of 2-D complex fluids. Understanding these systems is important both from fundamental point of view and their relevance in food, cosmetics, and pharmaceutical industries, where a whole class of soft material systems like particle stabilized emulsions and foams are ubiquitously present in a number of formulations. Previous studies on monolayers of polystyrene latex suggest that they exhibit wide range of structural morphologies ranging from 2-D crystals to exotic structures like dendrites and fractal like aggregates. The forces that determine the interaction of these particles are electrostatic forces, capillary forces and short ranged van der Waals forces. Simulations and experiments suggested the morphology of the monolayers can be tuned by parameters such as contact angle, surface charge, particle density, size and shape of the particles.

When the monolayer contains monodisperse particles it is possible to tune particle-particle interaction and hence the monolayer structure by the addition of salt or surfactant. Such aggregated 2D suspension are viscoelastic and effect kinetic stability of emulsions. In the present study, we use a binary mixture of oppositely charged polystyrene latex particles of two different sizes and systematically explore the effect of surface coverage and number ratio on the structures they form at decane-water interfaces. We adopt two protocols for spreading the particles. In the first method, the particles are premixed and then spread at the interface while in the second, the particles are added to the interface in a sequential manner. We observe the formation of structures ranging from hexagonally close-packed 2D crystals to fractal like aggregates to segregated domains of oppositely charged particles. Results also suggest that parameters like spreading protocol and relative number ratio of particles has significant effect on the structure of the monolayer. Evolution of monolayer structure with ageing was also investigated. Interfacial properties like surface moduli and surface viscosity of these monolayers were determined using a bicone geometry attached to a controlled stress rheometer. The results suggest that, for a given surface coverage, the monolayers containing oppositely charged particles exhibit higher modulus compared to the monolayer containing particles of single type.

**Key words:** *particle monolayer, fluid fluid interface, charged particles, interfacial rheology, fractal aggregate, bicone geometry, polystyrene latex.*

**Session 17.**  
**Rheology of Melted Metals and Glasses**

## **System Li,Na,K,Rb,Cs,La,Pu||F for the Generation IV Nuclear Technology: T-x-y Diagrams**

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Fluoride systems have been described as one of the possible fuels and coolant for molten salt reactor application. Besides the lowest melting point another criterion is the same important for the salt fuel: a concentration of fissile material. To optimize the molten salt fuel parameters the computer models of phase diagrams (PD) for the fluoride systems are to be built.

Computer model is based on the detailed geometrical description of a diagram: points of invariant equilibria, curves, surfaces, phase regions. As a result it permits to use PD as an instrument to solve different applied tasks of heterogeneous design and physics-chemical analysis.

The LiF-KF-LaF<sub>3</sub> system can be considered as a prototype of the possible fuels for molten salt reactor application.

The kinematical method is used for the unruled and ruled diagram surfaces simulation. In this case, a diagram surface is presented as the pseudo-ruled one, that is, a space curve, given by the interpolation polynomial, moves along the analogously given directing curves.

Data for binary systems LiF-KF, KF-LaF<sub>3</sub>, LiF-LaF<sub>3</sub> concerning the melt temperatures of initial components (LiF, KF, LaF<sub>3</sub>), coordinates of binary eutectics and peritectic and compounds (KF·LaF<sub>3</sub> and 3KF·LaF<sub>3</sub>) are used as initial data at PD simulation. The form of binary curves (i.e. curve convex downwards or upwards) are taken into consideration while the liquidus and ruled surfaces simulation. The coordinates of ternary eutectic, peritectic and two quasi-peritectics also was given as initial data.

As solid phase solubility in this system is not present, the solidus and solvus surfaces are absent. As a result the given PD includes five surfaces of primary crystallization, four horizontal complexes at the temperature of invariant points, and two vertical planes of triangulation. The surfaces curvature can be corrected according to the experimental data or thermodynamic calculation results. Considered phase diagram of LiF-KF-LaF<sub>3</sub> system includes a one-phase region L, seven two-phase regions and eleven three-phase regions. The elaborated phase diagrams computer models makes possible to study fluoride and other systems and can be used at the design of materials employing as possible fuel components for molten salt reactor.

**Keywords:** 3D and 4D of phase diagram, fluoride systems, molten salt reactor

## **Data Correction for Lead-Free Solders**

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3D computer model of phase diagram may be used for searching and correction of mistakes in its projections and sections, designed by thermodynamic methods. It may be confirmed on the phase diagrams Ag-Bi-Sn, Au-Bi-Sb, Ag-Cu-Sn in the COST 531 Project (Dinsdale A., Watson A. et al., Atlas of Phase Diagrams for Lead-Free Soldering. Czech Rep., Brno: Vydavatelství KNIHAR, 2008, Vol. 1). Twenty T-x-y diagrams of this Atlas have been created using the database in conjunction with commercial software Thermo-Calc, MTDATA, PANDAT. However some sections of these diagrams have some doubtful details, which may be explained only by special 3D models.

The system Ag-Cu-Sn isopleth A-(0, 0.1818, 0.8182) has two neighbor 2-phase regions, that breaks the adjoining phase regions rule. There is a problem with the same system on the isotherm 221C: it includes the 3-phase region which really exists in the interval 219.8-217.5C and can not appear at this temperature.

There are some questions to isotherms and isopleths of the system Au-Bi-Sb too. For instance, the only homogeneous region of solid solution Bi(Sb) exists at 240C in the binary system Bi-Sb, but it can be seen the sections of 2-phase regions Bi(Sb)+AuSb<sub>2</sub>, L+Bi(Sb), Bi(Sb)+Au<sub>2</sub>Bi on the section at the same temperature of the ternary system Au-Bi-Sb and all of them adjoin to the binary system Bi-Sb. While compounds Au<sub>2</sub>Bi and AuSb<sub>2</sub> do not belong to the system Bi-Sb. The 3D model helps to find two surfaces and two phase regions in sections of the Au-Bi-Sb T-x-y diagram, missed in the Atlas.

Quasi-peritectic reaction  $L+R_1 \rightarrow A+B$  is denoted by the letter D as the degenerated one in the Atlas invariant reactions table of the system Ag-Bi-Sn=A-B-C, where R<sub>1</sub> is one of two binary compounds in the system Ag-Sn. However, according to given in the Atlas temperature row, peritectic reaction  $L+A \rightarrow R_1$  proceeds the reaction D, and eutectic reactions  $L \rightarrow A+B$  and  $L \rightarrow B+R_1$  come after it. So, the reaction D is the peritectic one. If to use given in the same table concentration coordinates D, A<sub>D</sub>, B<sub>D</sub>, R<sub>1D</sub> of the isothermal complex vertexes, which correspond to participants of the reaction D, then the point D belongs to the triangle A<sub>D</sub>B<sub>D</sub>R<sub>1D</sub>. It says about eutectic character of four-phase reaction D. Hence, the invariant phase reaction D and univariant reactions  $L+A \rightarrow R_1$ ,  $L \rightarrow A+B$ ,  $L \rightarrow B+R_1$ , proceeds or follows it, require additional investigations.

**Keywords:** *Ag-Bi-Sn, Ag-Cu-Sn, Au-Bi-Sb, lead-free solders, computer simulation, 3D model, phase diagrams*

## **A novel viscosity model and database for molten slags**

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Based on the thermodynamic associate species model, a novel viscosity model and database are currently being developed for the system SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-CaO-MgO-Na<sub>2</sub>O-K<sub>2</sub>O. The modified Arrhenius viscosity model, employed in this database, is a structurally-based model, in which the associate species link the viscosity to the internal structure of molten slags. The model is capable of predicting the viscosity over a wide range of temperature and composition by using only one set of model parameters, all of which have clear physico-chemical meaning.

In silicate melts, the structural unit, i.e. the silica tetrahedron, can form a chain structure, a ring structure, and a network structure, which result in an increase in viscosity. Due to these changes of structure, i.e. silica polymerization, the viscosity can range over 14 orders of magnitude as a function of silica composition. The current viscosity model gives an effective consideration of the silica polymerization. Thus, one of the challenges of the viscosity behavior, the so called lubricant effect, can be described very well.

Another challenge, the amphoteric or charge compensation effect, can also be described very well by relying on the associate species MAI<sub>2</sub>O<sub>4</sub> and MSi<sub>2</sub>Al<sub>2</sub>O<sub>8</sub> (with M = Mg, Ca) in systems containing alkaline earth oxides or AlkAlO<sub>2</sub>, AlkAlSiO<sub>4</sub>, and AlkAlSi<sub>3</sub>O<sub>8</sub> (with Alk = Na, K) for the systems containing alkali oxides. Furthermore, the viscosity maximum does not always occur at the ideal composition, in which the ratio of Al<sub>2</sub>O<sub>3</sub> and MO respectively Alk<sub>2</sub>O equals one, which is consistent with experimental data available in literature.

**Keywords:** *viscosity, associate species, thermodynamic modeling, structure, molten slags*

## **Session 18.**

# **Rheology of Nanofluids**

## **Rheological behavior of screen printing pastes based on Barium Titanate nanopowder**

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The miniaturization tendency of electronic devices leads to the necessity of downsizing multilayered ceramic capacitors simultaneously with their capacity increasing. The specific capacity of the multilayered ceramic capacitors can be increased by thickness decreasing of ceramic and electrode layers and increasing number of electrodes. Thickness of metallic and dielectric layers can be decreased to 100-200 nm by using nanosize particles and new techniques of layer manufacturing. Screen printing technology is more promising one for thin dielectric layers obtaining, but film roughness and evenness are determined by the rheological behavior of screen printing pastes. Thus, investigation of rheological behavior of screen printing pastes based on BaTiO<sub>3</sub> nanopowder is of great importance.

Screen printing pastes have been prepared using BaTiO<sub>3</sub> nanopowder with mean particle size 20 nm, ethylcellulose as binder and terpeneol as solvent.

All observed pastes have pseudoplastic behavior. Solid content increasing in pastes with invariable binder content leads to raising of the paste viscosity at all shear rate range and shifting of flow curves to lower shear rate volumes. In this case flow curves shifting approximates pseudoplastic pastes behavior to plastic one. In addition, thixotropy degree lowering is observed that in some cases resulted in the changing thixotropic pseudoplastic behavior in pseudoplastic and rheopexic pseudoplastic ones. Ethylcellulose content decreasing leads to decreasing paste viscosity usually but sizeable viscosity increasing was observed at same solid/binder ratio in paste.

Generally it was determined that ethylcellulose had considerable affinity to BaTiO<sub>3</sub> nanopowder: low solid content changing led to sizeable paste densifying. This leads to solid content decreasing of nanopowder in paste from 50 – 70 % wt. that are typical for submicron powders to 10 – 15 % wt. Rheological behavior changing are associated with solid-binder structure changing in paste. At some BaTiO<sub>3</sub>/ethylcellulose ratio bonds between suspension parts are damaged, ethylcellulose molecule unrolls and all its functional groups bond with barium titanate powder. This is attended by the changing of thixotropic pseudoplastic behavior in pseudoplastic one. In the case of rheopexic pseudoplastic pastes, possibly, each barium titanate particle bonds with the several ethylcellulose molecules and formation of 3D-structure is observed. Damaging of this structure under shear stress/shear rate increasing leads to formation of disrupt bonds which generate additional paste densifying.

**Keywords:** *screen printing, pastes, shear thinning fluid, pseudoplastic fluid, thixotropy, thixotropy degree, rheopexy, shear stress, shear rate*



## **Rheological Properties of Silica Based Shear Thickening Fluids (STFs)**

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Shear thickening phenomenon is a non-Newtonian fluid behaviour that covers the increment in viscosity with increasing applied shear stress or shear rate. Shear thickening fluids (STFs) are concentrated colloidal suspensions which mainly consists of nano-sized particles distributed in a medium fluid. Rheological behaviour of STFs are receiving great attention in recent years due to their potential applications such as within liquid armor, suspension equipments and vibration damping systems.

In this study, STFs were prepared by ensuring the distribution of fumed silica nano-particles (90-250 nm) in polyethylene glycol (PEG) with the help of an ultrasonic disperser. Silica concentration range was set as 5-30 wt% in PEG that has varying molecular weights (200, 300 and 400 g/mole) to observe the effect of silica concentration and PEG molecular weight on steady and dynamic rheological behaviour of STFs.

Rheological behaviour of STFs and microstructural features of nano-sized silica particles were characterized by a TA AR2000ex rheometer, scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. In addition, thermal properties of PEG's were analyzed by differential scanning calorimetry (DSC).

Based on the experimental results, viscosity change becomes much more drastic with increase in silica particle concentration. This increase in value of viscosity indicates an incredibly high shear thickening effect which makes the fluid much impact resistant. On the other hand, PEGs having various molecular weights show slightly different results at the same silica concentration value.

**Key words:** *Shear thickening fluids, nano particles, rheological behaviour, characterization*

## **Improvement on viscosity of silica based nano-suspensions.**

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Nanofluids are engineered by suspending metallic or nonmetallic nanoparticles in heat transfer fluids such as water, oil, diesel, ethylene glycol, etc. Experiments have shown that nanofluids have substantially higher thermal conductivities compared to the base fluids. Since the agglomerate sizes can significantly impact the thermophysical properties of nanofluids and lead to a different heat transfer performance, the preparation of stable sols is of great importance. Viscosity of nanofluids is important from an industrial point of view as the viscosity has in fact an important effect on the required pumping power thereby reducing the overall benefit of a higher thermal conductivity fluid. In laminar flow, the pressure drop is directly proportional to the viscosity. Furthermore, convective heat transfer coefficient is also influenced by this parameter.

In this work, various parameters effecting stability and viscosity of silica nano-fluids were investigated. More precisely, concentration, formulation, sonication time, temperature and pH were varied for aqueous sols prepared using commercial fumed silica having different particle. Zeta potential, viscosity and the mean particle size measurements as a function of pH were performed in absence of dispersant. Finally, the stability of the nanofluids was also studied by Ultraviolet–Visible (UV–Vis) spectroscopy method following the investigated suspensions during a period of time. The results show that a combination of the knowledge about the chemistry of base fluid and nanoparticle suspensions lead to stable silica nanofluids without any stabilizers. The improvement on the stability and viscosity of the suspensions in terms of the suspension preparation methods and the ways to enhance thermal conductivities compared to the base fluids was considered/analysed.

**Keywords:** *nanofluids, stability, viscosity, particle size, zeta potential.*

## **Rheological properties of the BaTiO<sub>3</sub> nanopowder slips for tape casting**

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Creating a modern, efficient materials of electric- and radio-technical purpose impossible without systematic scientific research in the fields of material science and new technologies. Especially, this applies to multilayer ceramic capacitors, for which the miniaturization becomes particularly important. Multilayer ceramics has many advantages in performance and reliability, cost and space saving. Tape casting – is an important technology of ceramic processing, which are widely used in the production of thin sheets of flexible film. Tape casting is often the best choice because of its efficiency, relative simplicity and low cost in comparison with other available methods. Study of rheological properties of suspensions with nanopowders is relevant due to changes in the nature of the flow curves of suspensions to dilatancy, which previously tried to avoid when using submicron powders.

BaTiO<sub>3</sub> powder with a particle size in range of 20-25 nm, obtained by using the technique of non-isothermal decomposition of barium oxalate tytanil was used to prepare the tape casting slip. Ethanol, low polar organic solvent and menton were used as the solvents. Polyvinylbutyral and Butvar were used as binders. Dibutyl phthalate was used as plasticizers.

From the results of measuring the flow curve and dependence of viscosity on shear rate shows that the vast majority of slips for tape casting characterized by pseudoductility-dilatancy nature of the flow. Lack of dilatancy on flow curve was observed only with slips in which as an organic component was used ethanol and PVB and dibutyl phthalate as plasticizer. Changing any of the components of the mixture resulted in the appearance of dilatancy and significantly decrease of the viscosity of the slip. Based on these results, we can assume that for slips of BaTiO<sub>3</sub> nanopowders with low viscosity it is typical to form an individual particle of barium titanate with binder, which begin to interact with each other only at high shear rate.

Adding of dibutyl phthalate plasticizer, on the one hand, caused some thickening of the slip, and on the other - in almost all cases this led to appearance or strengthening of thixotropy. Instead, adding polyethylene glycol cause almost no effect on the rheological properties of the slip.

Effect of solvent type was observed only by change of the viscosity of the system at low values of shear rate: use of less polar solvents in the suspension increased the viscosity of slip.

**Keywords:** *Tape Casting, rheology, nanopowder, BaTiO<sub>3</sub>, slip, thin films, multilayer ceramic capacitors, flow curve, dilatancy, viscosity*

## **Rheological characterization of polyanionic cellulose(PAC) solutions**

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The rheology of aqueous solutions of polyanionic cellulose (PAC) at high molecular weight was investigated using a controlled stress rheometer. Several rheological measurements: viscosity measurements, creep compliance tests at a constant low shear stress and oscillation experiments have been performed. The concentrations ranged by weight from 0.01 to 2.5% of PAC. It was found that the aqueous solutions of PAC do not exhibit a yield stress, the flow curves of PAC over a wide range of shear rate (0 to 1000 s<sup>-1</sup>) could be described by the Cross model and the Carreau-Yasuda model. The critical concentrations of polymer  $c^*$  and  $c^{**}$  have been estimated. The dynamic moduli, i.e., storage modulus ( $G'$ ) and loss modulus ( $G''$ ) of the polymer have been determined at frequency sweep from 0.01 to 10 Hz. At polymer concentration above 1%, the modulus  $G'$  is superior to  $G''$ . The relationships between the dynamic moduli and concentration of polymer have been established. The creep-recovery experiments demonstrated that polymer solutions show important viscoelastic properties of system water-PAC when the concentration of the polymer increases.

**Keywords:** *Polyanionic cellulose, viscosity, creep, oscillation, Cross model*

## **Polymer Nanostructure Fabricated by using Nanoimprint process for OLED Applications**

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The power efficiency of organic light emitting diodes (OLEDs) is a critical issue in various practical applications. In a typical OLED, the light extraction efficiency is only about 20-30%, and most of the light is lost in the waveguide modes. The light extraction efficiency can be improved using various methods such as random microstructures, refractive index control, micro-cavities or two-dimensional (2D) photonic crystal (PC) structures.

In this study, we report on nanostructured OLEDs fabricated on polymer based nanostructures to enhance the light extraction. The nanostructures are fabricated using ultraviolet (UV)-NIL process at room-temperature and low-pressure. When there is no planarization layer coated onto the nanostructure, the OLED layers are deposited in such a manner that each layer follows the patterned surface of the previously deposited layer. This accounts for the formation of a multi-layer nanostructured corrugated (simply corrugated OLED). On the other hand, photonic crystal (PC) slab is applied for OLEDs are fabricated.

The lattice constant and diameter of the rods for corrugated OLEDs are 530 nm and 265 nm, respectively, and the heights of the rods are 15 nm, 50 nm, 65 nm, 80 nm and 100 nm. 49% enhancement of the light extraction with respect to the current density was obtained from the device having the corrugation depth of 50 nm.

On the other hand, the OLEDs fabricated on the nanosubstrates with the rods higher than 65 nm showed cracks in the IZO layer and voids at the interface between the IZO and the organic layers. Void formation leads to unstable current distribution, resulting in a considerable decrease in the light emitting efficiency.

In addition, The OLEDs integrated on PC structures are fabricated and evaluated. In order to predict the performance of the proposed device, a three-dimensional finite difference time domain (FDTD) simulation was performed. The roles of corrugated metal mirror and nanostructures on the efficiency enhancement were examined according to refractive index difference.

**Keywords:** Nanoimprint, Polymer nanostructure, Extraction, Photonic crystal, Surface plasmon,

## **Rheology Characteristics of Ceramic Inkjet Inks Based on Barium Titanate Nanopowder**

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A multilayer ceramic capacitors with high capacity and small sizes are in demand nowadays. The capacity of ceramic capacitors can be increased through thinning dielectric layers, what is possible only with using nanomaterials. Developing fundamental approaches to formulating technologies of functional materials with required properties and thorough rheological analysis fabricated stable suspensions are the most urgent problems of inkjet printing of ceramic inks. The rheology of the ink is of great importance for its performance during jetting and spreading on substrate. In addition proper selection optimal rheological characteristics of printing material is very important for manufacturing thin films. Rheological behavior is affected by many parameters such as solvent composition, presence and concentration of dispersant and pigment.

The ink for this application has been produced by two steps. At first, barium titanate nanopowder has been milled one hour with dispersant in planetary-type mill to obtain adsorbed layer on the surface of particles and then solvent has been added and milled hour again.

Rheological behavior matching which response commercial inks was the main criterion for selection ink composition. Rheological characteristics investigation of commercial samples shows that inks have dilatant flow and viscosity 3.5 mPa·s.

The ink vehicle was ethyl alcohol. In order to achieve a stable dispersion dispersing agent has been added. Solutions of dispersant show dilatant flow and viscosity increases from 2 mPa·s for pure ethanol to 24 mPa·s for pure dispersant. Extension quantity of dispersant resulted in more sloping growth of viscosity curves. Dispersant concentration in alcohol solution can reach 20 wt% and optimal concentration is 10 wt% and viscosity of the solution is 3 mPa·s.

Based on optimized concentration has been investigated influence different types of dispersants on rheological behavior of suspensions. As dispersant has been used 1,3-propanediol, ethylene glycol and tetraethylene glycol. All suspensions have similar rheological curves. This value of viscosity of liquid vehicle allows to stabilize only 5 wt% of functional material. Viscosity of ink increased to 4.5 mPa s.

**Keywords:** *rheology characteristics, inkjet printing, ceramic ink, barium titanate, dilatancy, dispersant, viscosity.*

**Session 19.**  
**Rheometric Measurement - Methods,  
Equipments and Errors**

## **Rheological characterisation of traditional ceramic suspension: difficulties and solutions**

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Traditional ceramic suspensions are fluid systems composed by a solid and a fluid phase. The liquid phase is commonly water while the solid one is a mixture of several natural and sintetic raw materials. As some of them have a weak chemical affinity with the liquid phase so they easily agglomerate together. Their densities are different: the liquid part being less than half as dense as the solid part.

Evaporation of the liquid phase, sedimentation and agglomeration of solid phase, chemical reactions among the different component of the suspension are the most important source of errors in the rheological characterisation of these system.

Hovewer, in spite of such difficulties, the determination of their rheology is fundamental from an engineeristic point of view. The planning and management of the plant as well as the reduction of defects in the finished products also depend on the control and knowledge of the rheological behavior of the suspensions.

In this work the main sources of error in the determination of the rheological characteristics of a ceramic suspensions are described and commented with examples. Whenever possible, countermeasures or the best approaches will be indicated.

**Keywords:** *ceramic suspension, rheology, source of errors, solutions*



## **Study of shape memory alloys and the phase transition by DMTA and DSC measurements**

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The problem of the production of shape memory alloys is the determination of the phase transition temperatures. The most important parameters of manufacturing these materials are the temperature range of shape memory effect and superelasticity. These parameters are difficult to detect by traditional material testing techniques.

There have been many examples of measuring transition temperatures of metallic materials by DSC method, which was technically designed for plastic specimens. This method has also become well known in metal technology for the last ten years. However, the DSC measurement does not give exact temperatures of transformation because of the accuracy of the system.

The goal of this research is the examination of the DMTA technique to measure the phase transition temperatures of shape memory alloys. The main unit of a DMTA is the furnace and the moving and the fix clamp. The applied temperature range was between room temperature and 200 °C. During the test, stress activation was applied and the strain was measured as function of temperature and frequency. The equipment is able to evaluate the complex modulus ( $E^*$ ) which contains the storage ( $E'$ ) and the loss modulus ( $E''$ ). These values describe the elasticity and the plasticity of the samples. The tendency of the change of modulus was determined by different samples. The results were compared to DSC measurement. The results of DMTA show that the storage and the loss modulus had local peak in the temperature region of the phase transition. It means that the samples showed viscoelastic behaviour.

After comparing the results of DMTA and DSC measurements, it can be seen that both techniques detected the phase transformation approximately at the same temperatures. These results make DMTA measurement a potential new technique in the investigation of metals. The benefit of introducing DMTA to the investigation of metals is that the DMTA is more sensitive than any other measuring processes. As a reason, the transition temperatures of metals can be measured very precisely. The other part of our research was studying a special kind of shape memory alloy, the Nitinol. It can be used as a material of new kind of actuator in prosthesis hands. These kinds of actuators are compact and silent, so they make it possible to build a moving prosthesis. To demonstrate what these actuators are capable of, a prosthesis hand was built based on our design.

**Keywords:** DMTA, DSC, phase transition, shape memory alloy, superelasticity, shape memory effect, complex module, storage module, loss module, Nitinol

## **Rheological Properties of Adding Inulin in WPC Solution in Different Physiochemical Properties**

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Physiochemical and rheological properties of Inulin in WPC solution were studied. Samples containing 2 % Inulin displayed shear thickening behavior by increasing shear rate and in a constant shear rate pH decreasing had no effects on shear rate. In pH=3 samples contain no salt, 1 and 5 mM salt WPC concentrated revealed shear thickening behavior by increasing shear rate. It also found that all these 3 solutions had the same viscosity and no yield stress. These solutions flow index were more than 1 showing their shear thickening behavior. The most flow index was related to solution with 1 mM salt. Solution containing 5mM with flow index close to 1 revealed a Newtonian fluid behavior. In pH = 5 the most and the least flow indexes were related to solution containing 1mM and 5 mM WPC concentrated salt respectively ( $n > 1$ ). In pH = 7 the flow indexes were close to each other, free salt sample just displayed yield stress. The most and the least flow index were same as pH= 5.

**Keywords:** *Inulin, Rheological Properties, Physiochemical Properties, WPC*

## **Rheokinetics dependencies of gel-forming compositions**

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Gel-forming compositions (GFC) are commonly used in oil and gas production industry for wet seal some parts of productive seam and stream-flow redistribution. These compositions are initially water-like liquids and can be easily pumped, but within oil-reservoir GFC turn into gel and lose their fluidity. The time of gel-forming defines the possible duration of pumping and depth penetration of GFC and is a very important for practical purposes. Technique of vibration viscosimetry (VV) is used to define the time of gel-forming. Oscillating probe is immersed into liquid and experiences mechanical resistance which is registered, and thus one obtains rheokinetics dependencies. After induction period "alpha" which is typically characterized by a horizontal or descending fragment of the diagram (for heating of the sample), the process of gel-formation is usually accompanied by viscosity increase. The latter reveals itself by monotonously increasing part "beta". However, viscosity cannot fully characterize the gel-forming process. An important property of gel is elasticity. The increase of elasticity module is accompanied by periodical fragment "gamma" in the time dependence of mechanical resistance - interference of acoustic waves radiated by the probe and reflected by a cell wall. At this point one of basic postulates of VV - the condition of an "infinite" medium is violated. But this phenomenon - dependence of experimental results on the size of measuring cell - can be used to determine the time of gel formation. Information is deduced by comparison of rheokinetic dependencies for identical samples obtained in measuring cells of various sizes. Being plotted on one graph in the same scale, these dependencies certainly coincide with each other in "alpha" and "beta" fragments and are distinctly different in "gamma" area - at the time of gel formation. Hence, it is reasonable to investigate dynamics of rheological properties for measuring cells of varying size and to interpret divergent area of acquired diagrams as the time of gel formation. Extremum positions and envelope curves for non-monotonic sections of these dependencies are shifted to more firm structure with an increase of measuring cell size. Keywords^ gel-forming composition, viscosity, elasticity, mechanical resistance.

## **Friction and phase transitions in ferroelectric device materials observed by Resonant Piezoelectric Spectroscopy**

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A novel experimental technique, resonant piezoelectric spectroscopy, was used to measure mechanical resonance frequencies and the damping of mechanical resonance modes. This technique uses the local piezoelectric effect of a sample to excite elastic standing waves. These waves are very sensitive to the development of microstructures such as polar nanoregions and tweed which dominate the paraelectric in ferroelectric device materials. The detection of elastic waves is achieved using a piezoelectric transducer attached at the end of an alumina rod, which is in contact with the sample and transmits any elastic signal emitted from the sample to the piezoelectric transducer. Apart from sample resonances, vibrations of the alumina rods are also observed. Considering that alumina only shows a small thermal expansion over a large temperature range and no phase transitions, the amplitude of these rod peaks gives an indirect measure of the average piezoelectric coefficient  $\langle d \rangle$  of the sample. In order to test our technique, we performed RPS and RUS measurements in partially ordered perovskite  $\text{PbSc}_{0.5}\text{Ta}_{0.5}\text{O}_3$ , which shows a ferroelectric transition at 298 K. While the minimum observed in the temperature evolution of the resonance mode frequencies reveals the phase transition temperature, RPS shows a coexistence regime in which paraelectric and ferroelectric phases coexist. Inside this coexistence interval we find strong competition between the advancing phase front between the cubic and rhombohedral phase and twinning in the rhombohedral material. Both pinning and unpinning of the phase front and propagating twin walls lead to energy transfer to the sample which is seen as inherent friction of the microstructure. This friction is then observed as broadening of the RPS line profiles. We will show for the first time that dynamical tweed structures and PNRs in the cubic phase have little friction while the characteristic ferroelectric friction occurs only when the microstructures evolve into twins and phase fronts at lower temperatures. Quantitative results will be discussed.

## **Examination of primer- and second-rate thermodynamic realignments of metals and metal-alloys, with application of DMTA equipmnet**

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The researcher work tends to get acquainted with the borders of the application of a high efficiency equipment in the matter of metals. With this equipment, which already testifies in the compound industry, a flasher query of some metal-related technological parameters (that's needed to heat treatment, or malleable shaping, or other posterior utilization) could be available.

The researcher team examines both the industrial metals (principally) as well as the high-tech materials and special metals (for example shape memory alloys) with DMTA equipment. Our current object is the monitoring of the recrystallization, in case of aluminium and copper alloys, and the investigation of the influential factors of this process. Those measurements' results, that the DMTA presented, had been compared with DSC and hardness analysis, and what is more with phase diagrams, whereby we try to conclude to the utility of DMTA, in matter of metal alloys.

The literature research added up to that, we are not the first to examine metals with DMTA. However, their (particularly Asian researchers) main object is to determine such attributions of the metals which can't be measured with the equipment, that the metal industry uses nowadays. Contrarily, we would like to write up the measurement method, whereby the features of the most frequent industrial heat treatment of the widely processed materials would be easily defined. Taking into account the chemical compound of the samples, we distinguished tin-brasses, brasses, and aluminium alloys.

In the interest of the taking place of the recrystallization, we had to work harden the samples. In order to monitor the effect of the work hardening rate, we applied two different rates: 50% and 75%. The heat treatment already took place in the DMTA, using different heating rates, where the specimens were loaded by inflection in the 2-point bending support with constant frequency and amplitude.

Compared to the DSC and hardness analysis with the DMTA curves, it seems, that almost all of the samples used by us revealed the recrystallization. A further consequence is that the appearance of recrystallization on the DMTA curves, significantly depends on the rate of the cold working, and less depends on the chemical compound of our samples. We succeeded in demonstrating the appearance of the loss factor, which refers to the fact that metals also possess viscoelastic, or similar to viscoelastic properties, interpreted in the set of complex numbers.

**Keywords:** DMTA, recrystallization, DSC, hardness

## **Improved OCT Velocimetry of Hard Sphere Colloidal Suspensions**

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Optical Coherence Tomography (OCT) originally developed for medical imaging is used in order to measure Doppler signals of light scattered off sheared fluids in order to determine flow velocities for given depths through the sample due to the use of a low coherence light source.

Velocimetry with this technique allows measurements of opaque materials with use of infra red light, with probe volumes of 3.4 pico litres, and a resolution of 9 microns.

Our improved technique overcomes the low frequency noise that made low shear rates difficult to measure by use of modulation techniques and optimised data acquisition.

The use of this technique is demonstrated with a study of Hard Sphere Colloids which have been previously observed to display shear banding.

## **Session 21.**

# **Rheology of Drug and Cosmetic Materials**

## **Optical microrheology measurements for determination of sol-gel transitions of chitosan hydrogels**

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Applied rheology has proven to be a versatile tool in material analysis, providing the information not only on bulk mechanical structure, but also the information on microscopic scale chemical structure.

Knowledge derived from mechanical properties can act as a key factor for biomaterials classification and quality control. In principle, any chemical reaction or physiochemical process is connected with changing the material structural properties therefore mechanical properties determination allows for easy assessment of such a reaction extent or process state.

In current work we will investigate the temperature dependent change of chitosan hydrogels physiochemical properties by determination of its rheological characteristics.

Acid solutions of chitosan salts show the ability of sol-gel transition under environment (temperature or pH) change. Such ability allows for using the chitosan and its thermogels as polymer matrix carrier of active bio-compounds like medicines.

However, for use of such carrier in vivo, the precise knowledge on lower critical solution temperature (LCST) is needed. That can be challenging for classical rheology as measurements are distorted by complicated transition dynamics and temperature control issues.

In our work we accept that challenge and show that main measurement problems can be solved by use of passive optical microrheology. The proposed technique relies in general on tracking the Brownian motion of fluorescent tracer particles suspended in investigated medium. The rheological parameters are calculated based on mean squared tracer's displacement evolution with time. In our case they will be used for determination of LCST temperature of sol-gel chitosan salts transition.

Our initial results show clearly that such an attitude can provide extremely efficient way for LCST temperature determination as the sample volume can be reduced by orders of magnitude compared with classical rheological techniques therefore the heat inertia and temperature control issues can be minimized. This, together with low equipment cost, time of analysis of order of seconds and the potential of automated sample handling proves that optical microrheology is a very versatile and powerful rheological technique.

### ***Acknowledgements***

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***Keywords:*** *passive optical microrheology, chitosan, sol-gel transition*



## **The influence of selected excipients on the rheological behaviour of chitosan based ocular pharmaceutical systems**

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**Aims:** Chitosan, a modified natural carbohydrate polymer, has received great attention in diverse scientific fields including pharmaceutical and biomedical research areas. Besides its low toxicity, mucoadhesiveness and biodegradability its special favourable rheological feature makes it a unique gelling agent for the design of ocular systems. Chitosan based (2.0 w/v %) ocular systems containing selected excipients were formulated in order to investigate the rheological influence of applied auxiliary materials. Rotational and oscillatory rheological properties of propylene glycol (1.0-20.0 w/v %), glycerol (1.0-5.0 w/v %) and castor oil (1.0-5.0 w/v %) containing chitosan gels were evaluated. The rheological behaviour of formulated ocular gels were compared before and after steam sterilization.

**Methods:** Rotational and oscillatory rheological measurements were carried out with Kinexus Pro Rheometer (Malvern Instruments Ltd.). Comparison of flow curves and oscillatory frequency sweep measurements in the linear viscoelastic region made possible the evaluation of rheological effect of selected excipients.

**Results:** In the applied concentration range the effect of propylene glycol among the selected excipients presents the most significant impact on rheology of chitosan formulations. Steam sterilization results in a reduced viscosity in most of chitosan gels. However, the presence of polyols appears to prevent the degradation of chitosan after steam sterilization.

**Keywords:** *chitosan based ocular systems, effect of polyol excipients, steam sterilization*

## **Nonlinear Rheological Behaviors of Different Types of O/W Emulsion-Based Mayonnaises in Several Shear Flow Fields**

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Mayonnaise is one of the most widely-used condiments made of vegetable oil, egg yolks, vinegar and seasonings. Among these materials, egg yolk contains lipoproteins, lecithin, and other phosphatides, which contribute to its emulsifying capacity, and egg yolk is the only permitted emulsifying agent in commercial mayonnaise. Mayonnaise is an oil-in-water (O/W) emulsion containing droplets of one liquid dispersed through a second liquid termed a continuous phase.

Due to the flocculation of adjacent oil droplets to form a network, mayonnaise is essentially a weak gel system. The strength of the interactions between oil droplets depends on the van der Waals attractions, which are balanced to some extent by electrostatic and steric repulsion. The quality of mayonnaise is dependent on the right balance between these forces.

Commercial mayonnaises can have an extremely multifarious composition based on various manufacturing formulas. Mainly, the quantity, number and amount of spices or other flavoring agents lead to differences in their quality. For this reason, it is difficult to set up the factors on which the quality depends.

Nowadays, consumers concern about the adverse health effects associated with over-consumption of certain types of lipids. This has led to a trend within the food industry toward the development of reduced-fat or reduced-cholesterol products. The creation of reduced-fat or reduced-cholesterol products with the same quality attributes as the original full-fat products has been a considerable challenge, and the food industry has been trying to produce these kinds of products (or substituting materials) for its continuous developments.

Rheology of mayonnaise is of great importance and its correct evaluation can provide valuable informations that can be used in quality control of commercial production, storage stability, sensory assessments of consistency and texture, design of unit operations, and knowledge of the effects of mechanical processing on the structure of the emulsions.

The main objective of this study is to systematically characterize and compare the rheological behaviors of different types (normal, reduced-cholesterol and reduced-fat) of mayonnaises using a rotational rheometry system both in steady shear and periodically small/large amplitude oscillatory shear flow fields.

**Keywords:** *Mayonnaise, Rheology, O/W Emulsion, Nonlinear Viscoelastic Behavior, Steady Shear Flow Properties, Oscillatory Shear Flow Properties*

## **Rheology and physical-chemical characteristics of the solutions of the medicines**

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The dynamics of the rheology of aqueous solutions of 10 plasma substitutes drugs and 10 antiseptic before and after mixing them with blood, plasma and pus, depending on the physico-chemical factors interact. Rheology of drugs studied in their movement within the flexible tubes (infuzators, injectors, injection needles, intravascular catheters, gastric, intestinal, and other probes and drains) and containers with biological fluids. It was studied the effect of the following factors: gravity, specific gravity, temperature, relative strength, internal pressure, carbonation, pH, osmotic activity, the total concentration of ingredients, the surface activity and the amount of medication.

It is shown that the rheology of solutions studied plasma substitutes drugs and antiseptic before and after mixing with blood, plasma or purulent masses mostly depends on the volume of drug solution, the temperature, pH (alkalinity), the osmotic activity, the total concentration of the ingredients, the specific weight and carbonation. It was found that the rheology of liquid and viscous and dense biological tissues improve drug having the following physicochemical characteristics: gipertermia, giperalkalization and gipergazation.

Found that the leader rheology improving drugs and biological tissues is sodium bicarbonate, hydrogen peroxide and carbon dioxide introduced into the pressurized drug (similar food carbonated beverages).

The data allowed to develop a new hygienic medicine designed to liquefy thick purulent masses with pleural empyema, peritonitis, rhinitis, sinusitis, conjunctivitis, osteomyelitis, and thinning of cerumen and dissolution of the lacrimal stones. New hygiene medicament comprises an aqueous solution of 0.5 - 3% hydrogen peroxide, and 0.5 - 10% sodium bicarbonate, carbonated with carbon dioxide to a pressure of 0.2 atm and heated up to +39 - +42 ° C [1,2,3].

**Keywords:** *new drugs, hygiene products, pus diluting agent, peritonitis, empiema pleura, osteomyelitis, septic wounds, physico-chemical properties.*

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## **Extensional Rheological Properties of Oral Nutritional Supplements**

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Oral nutritional supplements (ONS) are dietary foods prescribed for patients suffering from malnutrition, patients at risk of malnutrition or patients who are not able to meet the nutritional requirements by normal food. ONS are available not only as liquids but also as powders or pudding.

Two proprietary formulations (Fresubin® Crème and Fresubin® Yo Crème) based on oil-in-water emulsions containing sucrose, milk protein, soya lecithin as emulsifier, maltodextrin, thickeners, vitamins and minerals, among other ingredients, have been rheologically characterized in terms of the shear rheology (steady state and dynamic tests) as well as extensional rheology. Shear rheological measurements were carried out in a stress-controlled rheometer (MARS Haake) using a parallel plate geometry, while the elongational tests were performed in an orifice extensional rheometer based on the pressure entrance approach. The steady state viscous flow revealed shear-thinning behavior obeying the Sisko's model. In regard to the dynamic tests, frequency sweep tests were carried out in the linear viscoelasticity region at a constant stress. A high ratio of the storage modulus to the loss modulus was noticed in the whole frequency range investigated, so that both samples exhibited strong viscoelastic characteristics. Finally, for the extensional rheological characterization the pressure hole errors due to viscoelastic effects were considered for the pressure drop in the orifice, so that the extensional viscosity of both formulations exhibited strain-thinning, following a power-law model in the strain rate range investigated. The ratio of the extensional viscosity to the shear viscosity (Trouton ratio) exceeded by far the value of 3 corresponding to Newtonian liquids.

**Keywords:** *extensional rheology, oral nutritional supplements*

## **Comparative Study of rheological behavior of montmorillonite and kaolinite: Application to the dermo cosmetics**

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Therapeutic activity of clay minerals (phyllosilicates) is controlled by physical, physico-chemical, rheological properties, as well as their chemical composition. Powders, clays used in cosmetics are applied to the external parts of the body in order to beautify or change the physical appearance, or to preserve the physico-chemical conditions of the skin. Gels clays exhibit characteristics intermediate between liquid and solid and rheological behaviors include dilatant behavior or pseudoplastic flow, the yield point, thixotropy and high viscosities. Smectite (clay type 2: 1) and kaolinite (clay type 1: 1) are the main minerals used for this purpose. The fundamental difference between the behavior of clay minerals is their performance when they are dispersed in polar solvents.

Kaolinites dispersions exhibit a dilatant behavior (the apparent viscosity increases with increasing shear rate) with values of viscosity (flow resistance expressed as the ratio between shear stress and shear rate) around 300MPa s, to 70% w / v dispersion of kaolin water. This rheological behavior is attributed to pockets of closed deflocculated clay particles, which during shearing results from contact and interparticle interaction. Morphology (spherical or flat tubular) greatly affects the viscosity of dispersions of kaolinite.

Other parts, dispersions flocculated solids in the case of clay minerals 2: 1 are typically pseudoplastic. This behavior is due to the progressive destruction of the structure of the system. Shear force increases with as the interparticle associations are broken, hence the greater tendency to flow. The structure of colloidal aqueous dispersions of clay mineral 2: 1 product their greatest common properties: Yield Point (resistance to breaking of the structure) and the thixotropy. After the clay mineral is hydrated, the three-dimensional network structure is quickly constructed by a marked increase in viscosity. Gradually as time passes, the remaining platelets are the length to fill the available sites of the structure, where the gradual increase in viscosity with decreasing speed.

**Keywords:** *montmorillonite - kaolinite - Rheology - cosmetic dermatology*

## **Rheology and Structure of Poloxamer 407 (Pluronic F127)**

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Poloxamer 407 (or Pluronic F127) is a triblock copolymer with central hydrophobic chain of poly(propyleneoxide) [PPO] and two identical lateral hydrophilic chains of poly(ethyleneoxide) [PEO]. Poloxamer 407 aqueous solutions show thermo-reversible properties, which have been a great attraction for pharmaceutical scientists and technologists in optimising drug formulations. Reverse thermal sol-gel transition and low toxicity have been the basis of research into the use of poloxamer 407 as a possible drug delivery system in human body. It has been considered for topical delivery of lidocaine and anti-cancer agents. In addition, poloxamer 407 has been studied as a potential vehicle for injectables by both the intramuscular and subcutaneous routes.

In spite of its paramount importance in pharmaceutical applications, little attempts have been made to investigate the rheological properties of poloxamer 407 during the past several decades. Moreover, most of these studies have been focused only on steady shear viscosity and sol-gel transition phenomenon. There remain much more important areas that should be investigated from a rheological viewpoint. This is a main motivation that we have performed a comprehensive study as to the overall rheological evaluation of poloxamer 407 solutions and gels in a wide variety of flow fields most relevant to its actual application conditions.

Rheological studies become particularly helpful when predictable relationships for the rheological properties of complex materials could be developed from a molecular architecture. In our study, structural analysis is additionally accompanied using small angle neutron scattering (SANS), and differential scanning calorimetry (DSC), in order to elucidate the relationships between rheological properties and material structure.

**Keywords:** *Poloxamer 407 (Pluronic F127), Rheology, Structure, Sol-Gel Transition, Thermo-Reversible properties, SANS, DSC*